Fabrication of Ordered Uniform Porous Carbon Material and Its Application in Gas Separation

by

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the requirements for the
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CERTIFICATION OF APPROVAL

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ABSTRACT

The **main purpose** of this work is to fabricate ordered uniform porous carbons and to test their usage in gas separation. The concerned **problem statement** includes irregular pores size distribution which will result poor selectivity during gas separation process. Therefore, the existence of ordered uniform network in porous carbon is important to overcome the poor selectivity issue. Porous carbons are remarkable due to the excellent absorption capability in their volume and surface. Ordered uniform porous carbons could be formed by template synthesis. Different template materials used for the template synthesis process will produce dissimilar porous carbons properties in term of specific surface area, pore diameter and pores distribution. The **scope of study** for this work concentrates on two main issues. First issue is the effect of template material's properties to the porous carbons fabrication. While another one involved application of the fabricated porous carbons in gas separation. The **methodology** of this work starts with preparation of carbon precursor. To be more specified, the preparation of carbon precursor includes synthesis of inorganic template and impregnate of the template with organic template. The impregnation of the templates means the precursor is polymerized. Subsequently, the carbon precursor would undergo carbonization process. The organic material is carbonized. The final stage of porous carbons fabrication is etching process. Through this process, the inorganic template is removed. Properties of the porous carbon are determined through characterization process using equipments such as X-ray diffraction (XRD) and Scanning Electron Micrograph (SEM). The application of fabricated porous carbons in this work could be proven by carbon dioxide, CO_2 gas separation using permeation cell.

ACKNOWLEDGEMENTS

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CHAPTER 1

INTRODUCTION

1.1. Background of Study

Good hydrophobic property, high specific adsorption surface area, ability to interact with molecules at their surface and bulk of the material, as well as good mechanical and thermal stability are among the main reasons for porous carbons being widely applied in chemical industry. The importance of the porous carbon could be proven in various applications such as gas separation, water purification, catalyst support and also electrodes for batteries and fuel cells [1, 2].

Pore size and distribution of porous carbons is one of the major factors that determine its applications. IUPAC has defined three categories of pores, namely micropore (pore diameter < 2.0nm), mesopore (2.0nm < pore diameter < 50nm), and macropore (pore diameter > 50nm). Micropores function well in adsorption of small gas molecules. For instance, micropores porous carbons are used in automotive exhaust treatment before the gas is released into environment. Mesopores structure is suitable to be applied in absorption of larger molecules such as absorption of H_2S and CO_2 from natural gas. In addition, mesopore structure has been widely developed even in electric double layer capacitors as well. Macropores carbons play as important role in absorption of bulky organic materials. For examples, macroporous absorption is commercialized in environment protection such as recovering and purifying benzene, chlorobenzene, phenol and other organics compound. [3]

Unfortunately, majority of the porous carbons produced are not in uniform ordered network. This condition will result low selectivity, meaning that, undesired gas to be separated from a gas mixture is not efficient. Lower selectivity indicates that amount of undesired gas is separated out lesser than the desired gas in a gas system.

1.2. Problem Statement

Irregular pore sizes, non-uniform porous carbon network, and disorder pores distribution are among the identified reasons that affect the selectivity of porous carbon especially in term of gas separation application. **Selectivity** could be defined as moles of desired product over moles of undesired product. Low selectivity occurred when the desired product exists in lesser amount as compared to undesired product. The main objective of uniform ordered pores is to provide a path that allows only a particular molecule to be separated from a gas system depending on the pores' diameter. Therefore, the fabrication of ordered uniform porous carbon network is essential in ensuring the good selectivity occurred in the gas separation process.

1.3 Problem Identification

Better reaction path, continuous improvement in term of heat and mass transfer, and efficient separation process are some good examples of critical demand from chemical engineering industry nowadays. These indirectly have lead to the growth of porous carbons due to their ability to interact with molecules not only at their surface but also in the bulk of the material. As mentioned before, porous carbons are widely used as industrial absorbent. They exist whether in microporous, mesoporous or even macroporous. Until today, there are so many methods and materials used in order to produce mesoporous carbons. Method examples are [4]:

a. Carbonization of polymer with thermally unstable components
b. Carbonization of polymer aerogels such as resorcinol-formaldehyde resins
c. Catalytic activation of carbon precursors in the presence of metals and organometallic compounds.

In concern, most of these mesoporous carbons fabricated using methods mentioned above sustain broad pores diameter. Generally, **broad pore sizes would allow smaller molecules to pass through them while larger molecules remain in the gas system**. To be more specific, pore sizes which are large and not uniform will have negative impact on the gas separation process. The negative impact raises when **both of the**

desired and undesired gas molecules could pass through the pores conditioned that the gas molecules are smaller than the pores' diameter. This situation has depleted from the main purpose of gas separation. In gas separation, **only desired molecules are supposed to pass through the pores while the undesired are trapped by them.** Pore size, pores network and its structural order are strongly determined by:

a. Template agent
b. Precursor

1.4 Significance of the Project

It is known that the problem of non uniform order porous carbon could be overcome by **template synthesis.** Template synthesis has been extensively applied in recent years. It could synthesize mesoporous carbons with uniform and interconnected pores. The variations that control the porous system are template agent and precursor. Therefore, it is important to know characteristics of template and precursor used. The characteristics could be molecular sizes, network structural and withstands temperature. Besides that, relation between both template agent and precursor should be understood well. Each of the characteristics will determine micropores, mesopores and macropores formed. The selection of each template agent and precursor could be done based on 9 essential criterions shown below:

i. Usage of porous carbon
ii. Type and sizes of pores required to produced
iii. Surface area of porous carbon per unit volume and weight
iv. Production amount of porous carbon
v. Mechanical and thermal stability
vi. Hazardous to safety and environment
vii. Complexity of porous carbon in fabrication process
viii. Capital investment
ix. Regeneration of porous carbon

1.5 Objective and Scope of Study

There are two main objectives in this project:

a. To fabricate, study and understand ordered uniform porous carbon network

b. To test for CO_2 gas selectivity in gas separation process using porous carbon

In beginning stage, a proper exploration on journals with regards to uniform ordered porous carbon is necessary for the author's understanding and identification of the important points. The important points cover clear and detail information on why uniform order porous carbons are produced, how to produce it, what conditions for its fabrication, as well as when and where the application of porous carbon.

There are three main general procedures in producing porous carbon, simplified as below:

i. Preparation of carbon precursor and template

ii. Carbonization process of prepared carbon precursor and the template

iii. Process removal of template

In second phase of this research work, the focuses would be given more on gas separation process. Observation, analysis and comparison would be done based on ability of uniform ordered porous carbon to separate Carbon Dioxide, CO_2 gas.

Scope of study for this research work is not just limited to the methods producing porous carbon, but it covers as well examples of topic shown below:

a. Finding the right methods of producing uniform order porous carbon

b. Searching for the factor controlling the pore size and pores distribution

c. Analyse and how to control the pore size distribution

d. Improvement characteristic of fabricated porous carbon

e. How to fully utilize fabricated porous carbon in the function of selectivity

The scope of study includes as well the techniques, understanding and operations behind the equipments involved:

a. Scanning Electron Microscope (SEM)
b. X- ray Diffraction (XRD)
c. Permeation Cell Unit

1.6 The Relevancy of the Project

Porous carbons such as activated carbon have been in fact applied in chemical industry since long time ago. Porous carbons exist as microporous, mesoporous or macroporous. Business oriented and competitive environment have always be the driving force for any chemical industry related company to move and improve further in term of chemical reaction, separation system and waste treatment system. It is a fact that chemical industry is a serious and crucial business. This can be proven by a situation such as: a small change in chemical process may lead to the lost or profit gained by a company. The lost and profit involve thousand, billions or more of investment. Hence, we should understand that the existence of any company in this world is to generate profit as much as possible besides provides the betterment for mankind within consideration of safety and environmental issues.

With reference to the condition above, a minor change of porous carbon's structure and uniform ordered condition will give such a huge impact in chemical process. **The demand for continues improvement in gas separation, water purification, catalyst support as well as electrodes for batteries in industrial world have basically support the relevancy of this research work.** However, in this report, the author would have the discussion of uniform ordered porous carbon limited to its fabrication process and usage in gas separation only.

Modification of porous carbon to pose uniform ordered network will result higher selectivity. It provides better condition in term of gas separation. Undesired gas molecules will be separated from the gas system with better efficiency. This research work functions as an experiment phase to fabricate the most suitable uniform ordered porous carbon which could be used for carbon dioxide, CO_2 gas separation. The author has decided to use 3 different types of template materials (silica, zeolite and activated carbon). All of the template materials are grind accordingly into 3 different sizes range (particle size< 63µm, 63µm < particle size < 125µm, particle size > 125µm). The purpose of using different templates is to compare resulted porous carbons in term of their structure formed, pores size, pores distribution as well as their capability in CO_2 gas separation. The separation of CO_2 gas has been decided as CO_2 gas is the by-product in any hydrocarbon oxidation process in chemical industry.

1.7 Feasibility of the Project within the Scope and Time Frame

This project involves with 4 main stages shown below:

i. Grinding of inorganic template into specific sizes (1 week)
ii. Fabrication of order uniform porous carbon network (3 weeks)
iii. Characterization of order uniform porous carbon network (3 weeks)
iv. Experiment the porous carbon produced in term of CO_2 gas separation (4 weeks)

Table 1: Proposed Time Frame for Project Progress

Week / **Stages**	**1** (25 -29 Jan)	**2** (1-5 Feb)	**3** (8-12Feb)	**4** (15-19Feb)	**5** (22-26 Feb)	**6** (1-5 Mar)	**7** (8-12Mar)	**8** (15-19Mar)	**9** (22-26Mar)	**10-12** (29Mar-23 Apr)
i		▨								
ii			▨	▨	▨					
iii						▨	▨	▨		
iv									▨	▨

This project is feasible to be completed within 12 weeks if the schedule upon is followed and obeyed well. The duration proposed for each stage has basically included carbon). All of the template materials are grind accordingly into 3 different sizes range (particle size< 63μm, 63μm < particle size < 125μm, particle size > 125μm). The purpose of using different templates is to compare resulted porous carbons in term of their structure formed, pores size, pores distribution as well as their capability in CO_2 gas separation. The separation of CO_2 gas has been decided as CO_2 gas is the by-product in any hydrocarbon oxidation process in chemical industry.

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ii										
iii										
iv										

This project is feasible to be completed within 12 weeks if the schedule upon is followed and obeyed well. The duration proposed for each stage has basically included the consideration of lab operation hours, availability of machine and public holidays. Overall, stage 2, stage 3 and stage 4 take longer duration. Stages 2 required plenty of time especially on sample drying and sample heating. While stage 3 consumed longer times due to standard procedural instruction which requires approval from related authorities before equipment booking could be done (Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD). This process might be delayed as well depending on the availability of the technician in-charged. The final stage, which is involvement of porous carbon in gas separation experiment, requires longest duration. It is time consuming due to the process of fabricating porous carbon in term of membrane form. The membrane formed will be used in the permeate cell unit for gas separation testing.

Note:

The laboratory operation hours are:

- Monday to Friday except for public holiday.
- 9.00am to 5pm (8 working hours)

CHAPTER 2

LITERATURE REVIEW

2.1 Template Synthesis

Fabrication of Uniform Ordered Porous Carbon involves general procedures shown below:

a. Grinding process: granular particles are grinded into desired sizes to be used as inorganic templates
   - Selection of suitable template material
b. Carbon precursor is prepared
   - The precursor is polymerized
c. Carbonization of carbon precursor
   - Organic material template is carbonized.
d. Etching process
   - removal of inorganic template

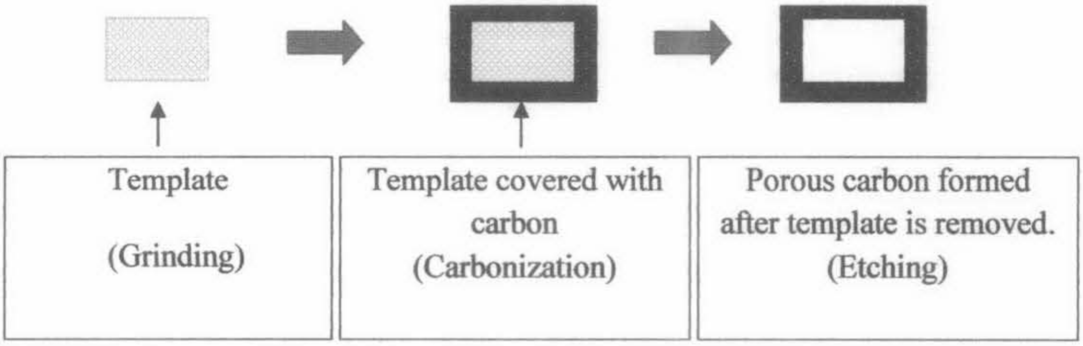


Figure 1: Concept of Template Synthesis Process

2.1.1 Synthesis of Nanoporous Carbon Materials Using Silica Nanoparticles as Templates.

Zakidov et al has used colloidal silica crystals as templates to synthesize the three dimensionally periodic with closed packed macroporous carbon materials. Silica crystal is a source of synthetic opal. In this method, the carbon precursor is formed by polymerization of silica opal plates with phenol resin. The carbonisation process for this sample is done in chemical vapour deposition using propylene gas. For the final step, Hydrofluoric acid is used to etch and remove the silica template. As a result, the porous carbon formed is totally inverse pattern of the silica template [7].

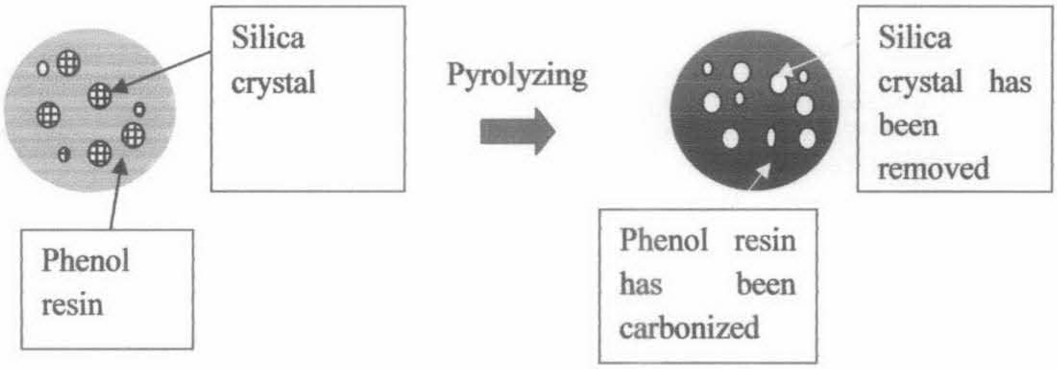


Figure 2: Theory of Porous Carbon before and after Carbonization Process

T. Hyeon and his research group have also conducted an experiment to produce nanoporous carbon. In their experiment, aqueous silica sol (nanoparticles) is used as template. While the carbon precursor, which are resorcinol and formaldehyde, is polymerized in the presence of silica sol solution (Ludox HS-40 silica sol solution, with average particle size of 12nm). After carbonisation of resorcinol-formaldehyde gel and silica composite, the etching process takes place. The silica template is etched, leaving behind nanoporous carbons, which designated as Silica-sol Mediated Carbon (SMC1).

Table 2: Properties of SMC1

Criterion	Properties
Pore sizes range	10 to100nm
Pore volume	> 4cm^3 g^{-1}
Surface area	1000 m^2 g^{-1}.
Pore size distribution	broad
Advantage	Good adsorption capacities for bulky dyes
Concerned issue	Broad pore size distribution (not uniform)

Broad pore size distribution of this porous carbon is due to the aggregation of silica nanoparticles during synthesis process [9].

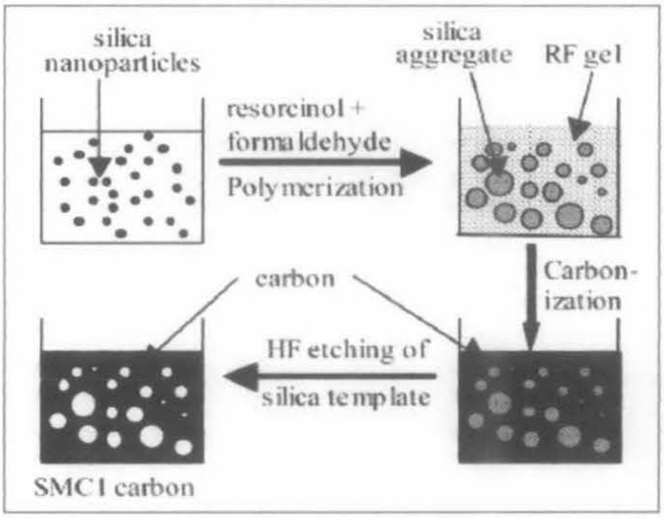


Figure 3: The Process Synthesis of Nanoporous SMC1 Carbon [1]

"Aggregation of silica nanoparticles during the syntheses process could be prevented by using surfactant-stabilized silica nanoparticles as templates", discovered by T.Hyeon and his research group. Template material such as cetyltrimethyl ammonium bromide (CTAB), a chemical which has cationic surfactant, is used to the stabilize silica sol nanoparticles. It prevents the aggregation of silica nanoparticles during the synthesis process.

Table 3: Properties of SMC2

Criterion	Properties
Pore sizes range	Uniform 8nm
Pore volume	$1.7cm^3g^{-1}$
Surface area	$1089m^2g^{-1}$
Pore size distribution	Averagely 12nm
Advantage	high pore volume and high surface area
Concerned issue	Poor pore connectivity

Fortunately, the poor connectivity issue is solved by T. Hyeon and his team as well. They use non-spherical rod-shaped or elongation silica template, producing nanoporous carbon which has good connectivity [10]:

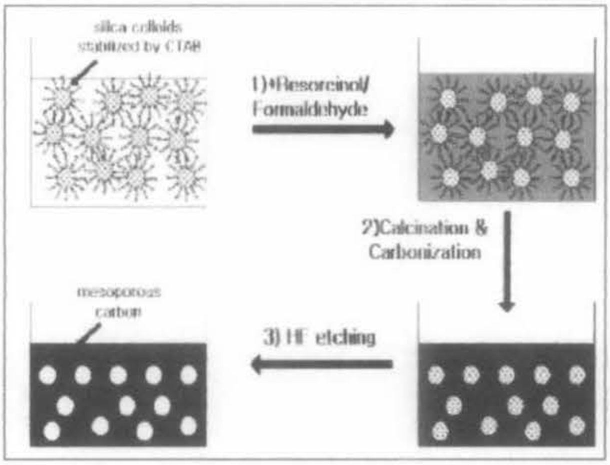


Figure 4: Synthetic Strategy for Uniform Nanoporous Carbons (SMC2) [1]

*This journal has basically **generated some ideas to the author (me) on how to overcome** the fabrication porous carbon's problems such as **aggregation and poor pore connectivity.** The author learned that the porous carbon uniform ordered could be improved and affected by material types of template. However, **it comes to author's concern on HF used during the etching process**. It should be substituted by Ammonium bi-fluoride or Potassium Hydroxide. These chemicals are less dangerous and less hazardous. Potassium hydroxide has high thermal stability and low melting point, which is safer to be used during experimental work.*

2.1.2 Synthesis of Ordered Microporous Carbons Using Zeolites as Templates

Generally, zeolites are known as aluminosilicate materials with ordered uniform sub-nanometer sized pores. Kyotani et al have fabricated microporous carbon using USY zeolite as the template. Before the carbonization process, carbon is introduced into the channels of USY zeolite by carbon vapour deposition (heating propylene at 700 °C to 800 °C). Furfuryl alcohol is injected in the zeolite's channels during carbonization process. As a result, micropores and mesopores formed in the porous carbon. Based on Kyotani, the carbonization occurred inside the channels of zeolite. It is proven by the morphology of microporous carbon particles which pose similar structure as zeolite template material [10].

Table 4: General Properties of USY Zeolite

Criterion	Properties
Pore sizes range	0.3 to 1.0 nm
Surface area	$2000 m^2 g^{-1}$

Kyotani and co-workers has improved the properties of the USY zeolite-templated porous carbon using ordered zeolite Y structure with two step method. Zeolite-poly (furfuryl alcohol) composite is heated at 700°C for 4 hours. It is followed by carbon deposition of propylene at 800°C for 4 hour. Kyotani described that the porous carbon obtained from this method showed an ordered zeolite replica structure. The present of the amorphous peak in XRD result has encouraged Kyotani and co-workers on finding a solution to treat it. Amorphous peak is undesired as it represents partial collapse of the carbon framework in the zeolite channels. This research group has eventually found the treating solution; by conducting the two step method in 900°C. Figure 5 and Figure 6 are HR TEM image of related porous carbon and XRD pattern zeolite Y and the resultant carbon material [11].

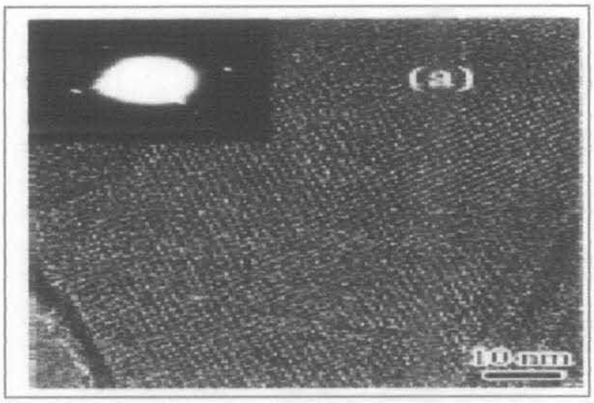


Figure 5: HR TEM image of carbon prepared in two- steps method at 900°C [1]

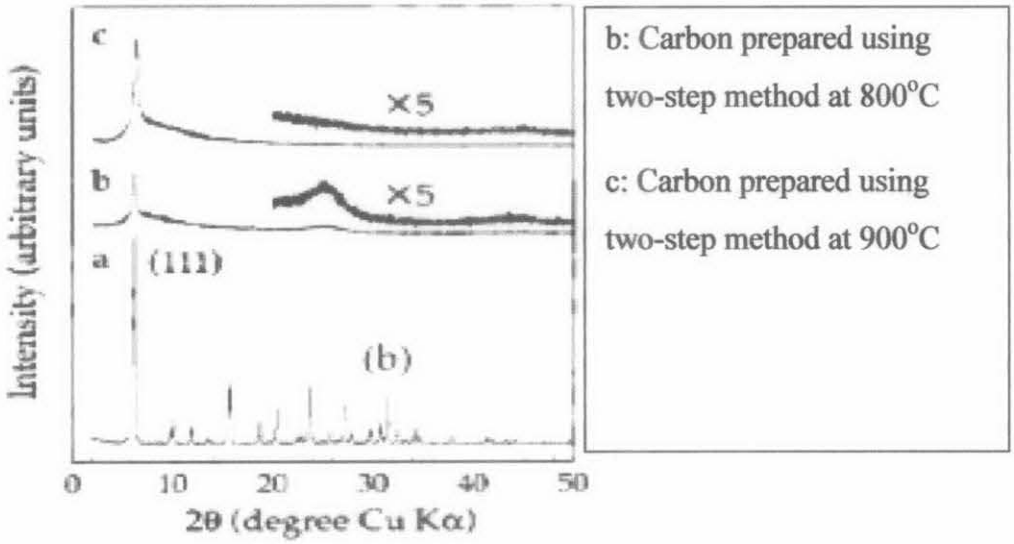


Figure 6: XRD Patterns of Zeolite Y and the Resultant Carbon [1]

The author (me) learnt that*, increase in temperature (100°C) in two steps methods could eliminate amorphous properties in porous carbons (zeolite-templated). Amorphous peak existed in XRD result is undesired as it represents partial collapse of the carbon framework in zeolite channels. Related to this method, the author would like to have a clarification on the* ***effect on porous carbon properties****,* ***if higher temperature*** *(more than 1000°C) is applied during two steps method. Moreover, it would be a bonus to the author if he could have a clarification as well on whether other template materials could apply this two step method.*

2.1.3 Synthesis of Uniform Mesoporous Carbon Materials Using Mesoporous Silica Materials as Templates.

MCM 48 silica material has three-dimensionally interconnected pore structures. The properties of MCM48 has led Ryoo group to deposit Aluminium in MCM-48 in order to generate the strong catalyst sites, called AIMCM-48.The strong catalyst sites are important for the polymerization of phenol and formaldehyde. The composite of AIMCM48-phenol is mixed with paraformaldehyde in autoclaves. The reaction of AIMCM48/phenol-paraformladehyde is allowed at 125°C. After carbonization, HF is used to wash away the MCM-48 frameworks. As a result, mesoporous carbon named SNU-1formed.

Figure 7: SNU-1 Mesoporous Carbon Properties

SNU-1 Mesoporous Carbon Properties
• Not real replica of MCM-48. (proven by XRD result in figure 10)
• Has undergone transformation to a new structural symmetry after the removal of silicate.

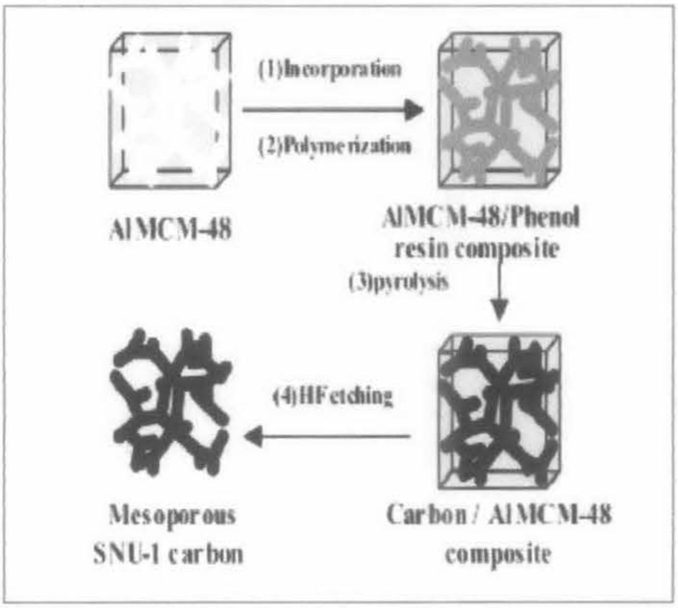


Figure 8: Formation of Ordered Mesoporous Carbon SNU-1 [1]

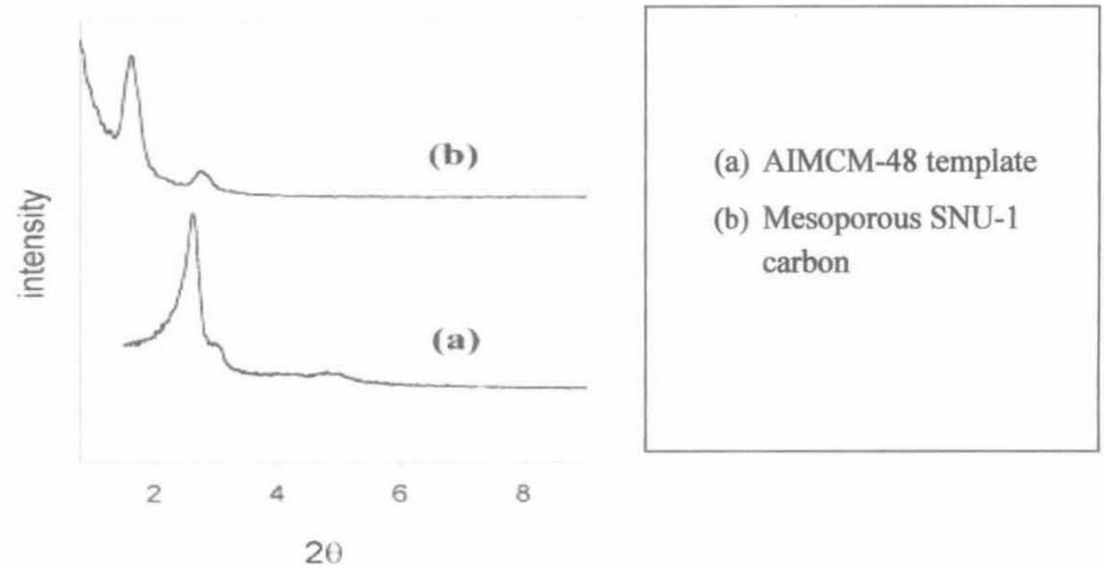


Figure 9: XRD Patterns of AIMCM-48 Template and Mesoporous SNU-1 carbon [1]

In this section as well, Ryoo et al have described the methodology of carbon molecular sieves (CMK-1). CMK is formed by carbonization of sucrose (carbon precursor) inside the mesopores of the silica molecular sieves MCM-48.The carbonization is done in the absence of inert atmosphere at 800 oC to 1100 °C. The MCM-48 template is removed using sodium hydroxide. Below is Figure 7, TEM image of CMK-1 and Figure 8, XRD patterns CMK-1 with its silica template MCM-48 during synthesis of carbon. [12].

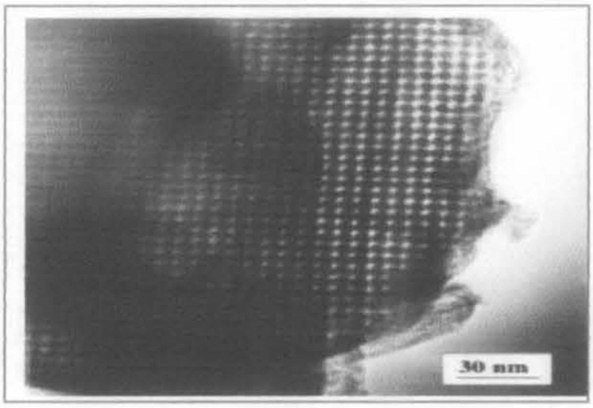
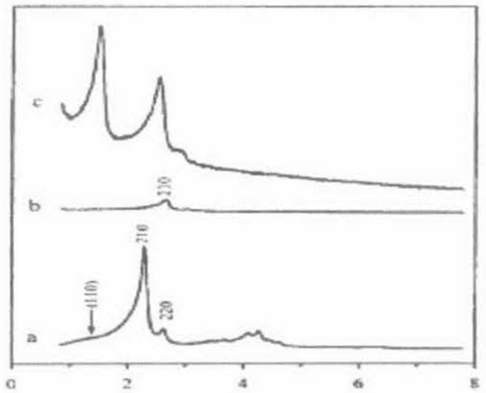


Figure 10: Transmission Electron Micrograph (TEM) of CMK-1 [2]



a: Mesoporous silica molecular sieve MCM-48,
b: MCM-48 after completing carbonization within pores
c: CMK-1 obtained by removing silica wall after carbonization

Figure 11: XRD Patterns during Synthesis of the Carbon CMK-1 and MCM-48 [2]

Different point of XRD peak for a, b and c indicate the CMK-1 has new ordered structure after carbonization process. Joo et al have proven the synthesis method could be controlled by altering the variables such as sucrose sulphuric acid ratio, Pyrolysis temperature, and the amount of sucrose. Shown below is the properties table of CMK-1.

Table 5: General Properties of CMK-1

Criterion	Properties
Pore sizes range	0.5-0.8 nm (micropores), 3.0nm (macropore)
Surface area	1380m^2/g.

In term of personal opinion, the ***author is impressed*** *with the* ***usage of silica material*** *due to its three* ***dimensional connected pore structure****. The author has come out with a* ***conclusion*** *after a comparison between zeolite and silica materials is done.*

- *Porous carbon formed using silica material did not retain its original template compared to zeolite.(SNU-1 porous carbon case)*

From the fabrication of CMK-1, the author was noted with the ***important of impregnation process****. It helps to load more carbon precursor into the template material. Through CMK-1 fabrication as well, the author was noted that the* ***synthesis process could be controlled by altering some variables*** *(especially carbon precursor).*

2.1.4 Direct Synthesis of Mesoporous Carbon

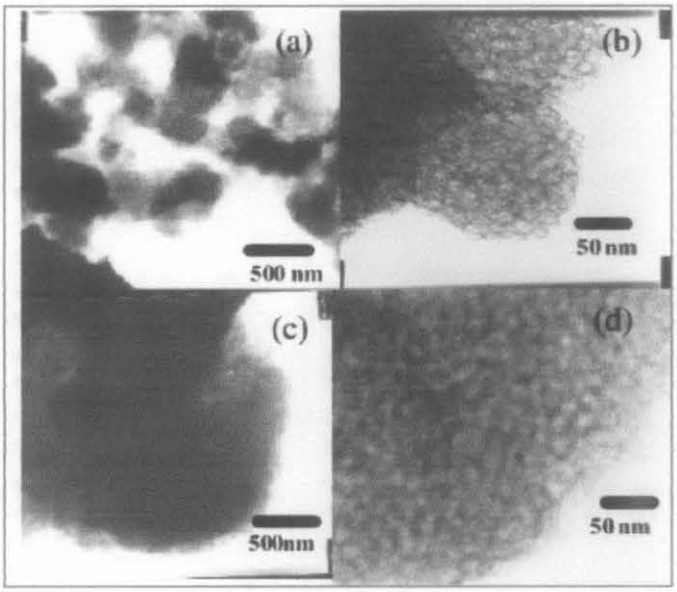
The waste of the production cost is taken into consideration each time the silica template is sacrificed at the end of the preparation of mesoporous carbons. Pinnavaia group has the solution related to mentioned issue. They have come out with a solution by producing hexagonally ordered mesoporous carbons using highly ordered mesoporous silica molecular sieves. In this method, sodium silica is used and as the silica precursor. Trimethylbenzene (TMB) is added in mesocellular siliceous foams (MSU-F), which acts as a synthetic solution.

Mesocellular siliceous foams (MSU-F) can be obtained also by using sodium silicate as the silica source under the near neutral conditions. The synthesized mesoporous carbons existed as hexagonal structure is due to the characteristic of mesoporous silica molecular sieves used in early stage. The present hexagonally ordered mesoporous carbons (C-MSU-H), poses two dimensional hexagonal structure. The pores sizes formed are approximated to be 3.9nm.

Under this category as well, Hyeon T and his group have developed a new route synthesize the mesocellular carbon by using cheap MSU-F silica as the inorganic template. Hydrothermal treatment is done so that MSU-F silica material has rich nanometer sized primary particles. MSU-F was aluminated and wetted with furfuryl acohol. It was later polymerized at 85°C under a static vacuum. After carbonisation and etching process, new porous carbon foam called C-nano-MSU-F formed. It is highly desirable as mesoporous materials due to its few hundred nanometres sized individual particles. The small individual size is important for the large molecules to approach the framework pores.

Table 6: C-nano-MSU-F Properties

Criterion	Properties
Pore sizes range	Cellular pores of 20nm, small mesopores 4nm
Advantage	Hundred nanometres sized individual particles is important for the large molecules to approach the framework pores.



(a, b) C-nano-MSU-F

(c, d) MCF- carbon

Figure 12: TEM Images C-nano-MSU-F and MCF- Carbon [1]

The author ***agreed with the fact of recycling the template used****. It was definitely one of the best ways in reducing porous carbon production cost. This method has produced a* ***unique characteristic mesoporous*** *.However, the* ***long and complicated multi-step template procedures have over shadowed its advantages****. Therefore, a shorter, simple and efficient route should be developed. Overall, this method has an advantage in term of consuming minimum level of cost production. However its drawback existed in term of time consuming for the template preparation.*

2.2 Conclusion of Literature Review

The author has included and stated his view at the end of each journal reviewed. The exploration of journal on various "porous carbon production methods" has helped the author's understanding in term of uniform ordered porous carbon fabrication process. Through literature review the author (me) has identified the strengths and weaknesses of porous carbon production method.

The **principles and conditions** that have guided author in selection of the most suitable porous carbon fabrication method to be applied in the experimental phase:

a. Production of uniform ordered porous carbon
b. Simple and less time consuming route of production/method
c. Porous carbon formed must exhibit high pore volume
d. Porous carbon formed must poses high surface area
e. Porous carbon formed must have good thermal and mechanical stability
f. Lower cost of fabrication

Based of the conditions shown upon, the author has come to final conclusion and selected **"Synthesis of Uniform ordered Mesoporous Carbon Materials Using Mesoporous Silica Materials as Templates"** as the method of producing porous carbon. The **carbon precursor** to be used is **mixture of sucrose, distilled water and sulphuric acid.** Due to the comparison purpose**, the author** would suggest **to have additional 2 templates materials, zeolite and activated carbon,** to be experimented during the fabrication phase. (Same experimental procedure as silica template is applied for other 2 templates through out the porous carbon fabrication phase)

The selection of the method mentioned is due to a few reasons such **as proven uniform ordered porous carbon** in the journal, **availability and lower cost of carbon precursor** used, **simple** and **less time consuming** production route. (Fulfil almost all of the principle/conditions mentioned)

.

Listed below are some of the improvement options that could be adopted for improvement of uniform ordered porous carbon fabrication.

a. Surfactant-stabilized silica nanoparticles as templates
  -to avoid aggregation of silica nanoparticles

b. Non-spherical rod-shaped or elongation silica template
  -to improve pore connectivity

c. Two steps method is done in higher temperature
  -to eliminate amorphous peak in XRD

d. Carbon precursor impregnation
  -to obtain higher loading of carbon precursor in template material

e. Control the synthesize process
  -could be done by some variables (especially related to the carbon precursor)

f. Hydrothermal process
  -to enrich nanometer sized primary particles.

Note: All of the options are referred to journals discussed upon.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

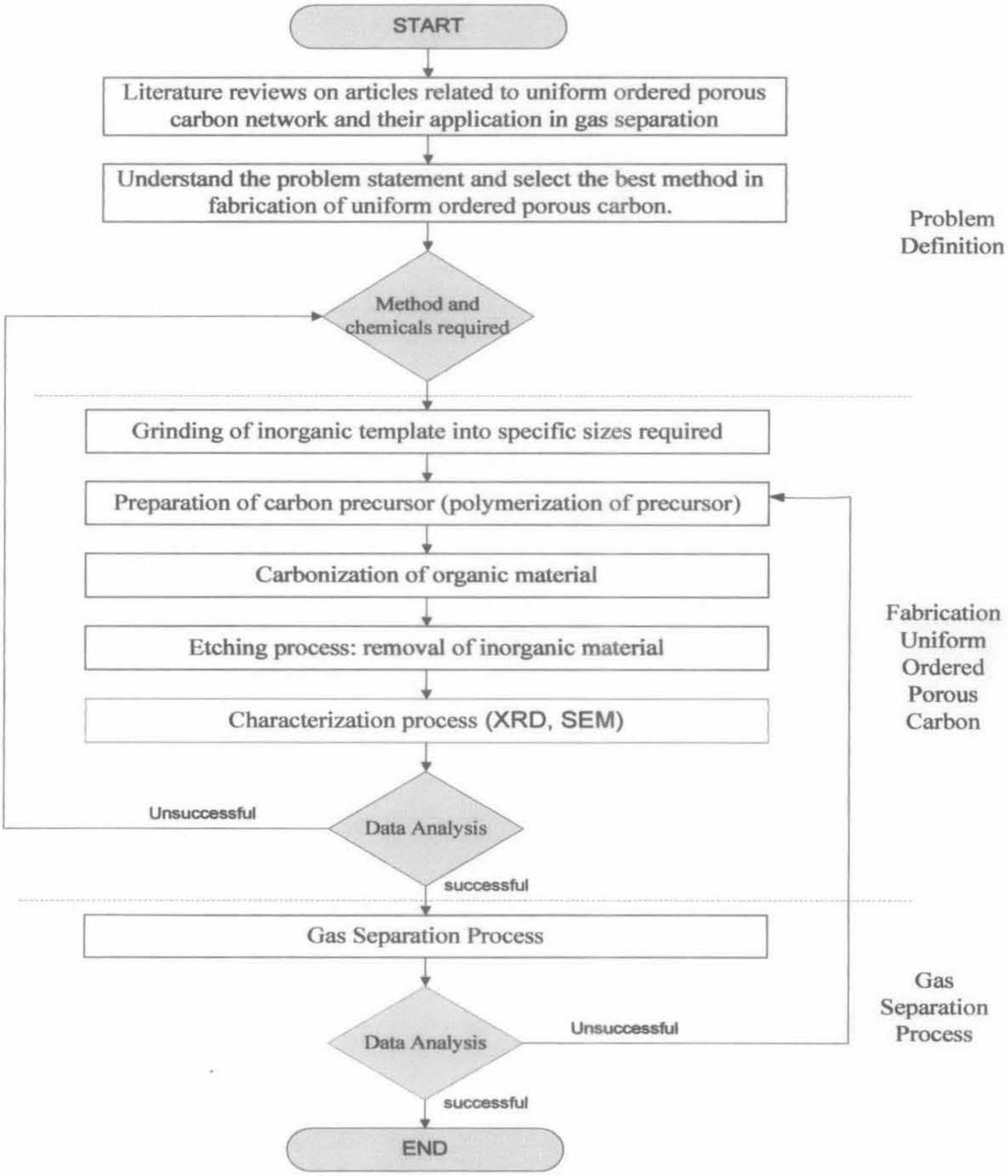


Figure 13: Research Methodology

3.2 Project Activities

Well established, organized and systematic methodology could help and also ease a researcher to manage and conduct experimental work in efficient manner. It enables a researcher to perform and complete planned tasks within the time and duration allocated. This research work could be divided into four major sections:

a. Problem definition
b. Fabrication of uniform ordered porous carbon
c. Gas separation analysis
d. Documentation

Further elaborations of sections a, b, c and d are shown below:

3.2.1 Problem Definition

i. Problem statement and the objective of this project should be clearly identified and understood. The understanding of problem statement, research objectives and scope of study could narrow down the variables existed in this experimental work. With minimum level of variables, better attention and focused could be performed in running experiment, thus, the objectives of this research work could be achieved.
ii. This research work must be relevant to any industry's demand and application to ensure the validity and its effectiveness in improving industrial's development.

3.2.2 Fabrication of Uniform Ordered Porous Carbon

i. Grinding process: granular particles are grinded into desired sizes to be used as inorganic templates
ii. Carbon precursor is prepared: the precursor is polymerized
iii. Carbonization of carbon precursor: organic material template is carbonized.
iv. Etching process: removal of inorganic template

3.2.3 Characterization Process

i. Porous carbon's pore distribution is viewed by using scanning electron microscope (SEM)

ii. Identification of porous carbon's structures by X-ray diffraction (XRD)

3.2.4 Gas Separation Analysis

i. The application of fabricated porous carbon in gas separation: tested with carbon dioxide gas, CO_2

ii. Data collected from this testing is plotted in to a graph for better understanding and analysis.

3.2.5 Documentation

i. Reference and citing are important to ensure the validity of this work.

ii. Each information, data, result and discussion of this research work should be compiled in organized and systematic layout for reference purpose in the future.

3.3 Key Milestone

Table 7: Key Miles Stone for final Year Project 2

Final Year Project 2 January 2010- June 2010													
No	**Detail/ Week**	**1-2**	**3**	**4**	**5**	**6**	**7-10**	**11**	**12**	**13**	**14**	**15**	**18-19**
1	Briefing and Updates on Student Progress	▨											
2	Project Work Commences			▨									
3	Submission of Progress Report 1				▨								
4	Submission of Progress Report 1 Marks					▨							
5	Submission of Progress Report 2						░						
6	Poster Exhibition/ Pre EDX							▨					
7	EDX								▨				
8	Submission of Final Report (CD)										▨		
9	Delivery of Final Report to External Examiner/ Marking By External Examiner											▨	
10	Final Oral Presentation												▨
11	Submission of Hard Bound Copies												▨

Table 8: Gant Chart for Final Year Project 2 January 2010 to June 2010

Final Year Project 2 January 2010- June 2010																
No	**Detail/ Week**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**18-19**
1	Briefing And Updates on Student Progress	▨															
2	Safety Briefing on Lab And Regulations	▨															
3	Grinding of inorganic template into specific sizes		▨														
4	Fabrication of Porous Carbon			▨	▨	▨											
5	Submission of Progress Report 1					▨											
6	Characterization of Porous Carbon						▨	▨	▨								
7	Gas Separation									▨	▨	▨	▨	▨			
8	Submission of Progress Report 2 (Draft final report)											▨					
9	Poster Exhibition/ Pre EDX											▨					
10	EDX												▨				
11	Submission of Final Report (CD softcopy)														▨		
12	Delivery of Final Report To External Examiner/ Marking By External Examiner															▨	
13	Final Oral Presentation																▨
14	Submission of Hard Bound Copies																▨

▨ Represents Work Progress

3.4 Equipment

This research work could be classified into three stages, mainly are fabrication of porous carbon, characterization of porous carbon and gas separation. Each of the stages requires different equipment depending on the purpose and objective of the process

3.4.1 Fabrication of Porous Carbon

3.4.1.1 Furnace

3.4.1.2 Oven

3.4.2 Characterization of Porous Carbon

3.4.2.1 Scanning Electron Micrograph (SEM)

3.4.2.2 X-ray Diffraction (XRD)

3.4.3 Gas Separation

3.4.3.1 Permeation cell

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Data Gathering and Analysis

After detail research and reference on journals, articles and other available sources, the author has finalised the procedures and steps involved in fabrication of ordered uniform porous carbon network. The steps could be divided into 4 mains stages as show below:

Stage 1: Grinding of inorganic template into desired sizes
Stage 2: Fabrication of order uniform porous carbon network
Stage 3: Characterization of fabricated order uniform porous carbon
Stage 4: Gas separation and analysis

4.1.1 Stage 1: Grinding of inorganic template into desired sizes

Inorganic template

a. Silica granular (Commercial)
b. Zeolite granular (Serum Triglycerides)
c. Activated Carbon granular (Commercial)

Equipment

a. Oven (60°C)

Apparatus

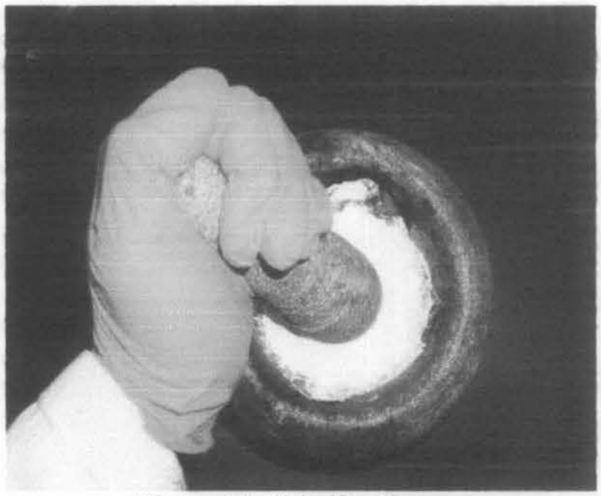
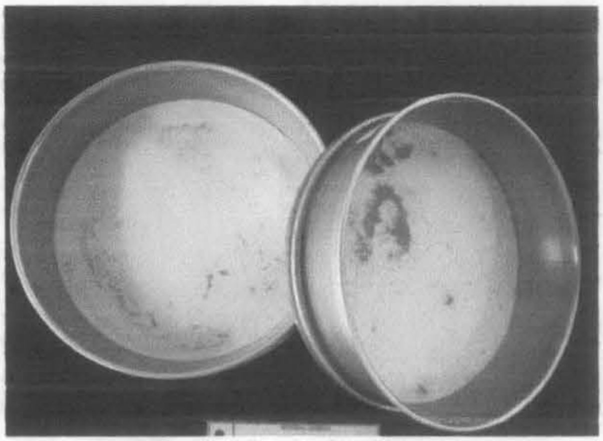
a. Grinding Stone (base: 9.5cm x 14cm height)
b. Sieving plate (Diameter pore size 63 μm and 125μm)
c. Electronic weigher (Company: Mettler Toledo)

Procedures (Experiment 1, Part A):

1. Total of 50gram silica granular is grinded for 45 minutes until solid powders are formed.
2. The sample is sieved with (250 µm pores diameter sieve plate) until 15gram of diameter D> 250µm sample is obtained. The sample is sieved again with sieve plate 125µm of pores diameter. Minimum of 15 gram powder diameter for each 125µm > D> 63µm and D<63µm are obtained again.
3. The sample is dried in 30°C oven for 1 hr to remove possible trapped moisture.
4. Procedures1-3 is repeated using zeolite and activated carbon.
5. At the end, there will be total of 9 samples prepared as shown below:

Table 9: Types of Inorganic Template and Particle Diameter

No	Inorganic Template / Particle Diameter	**D < 63µm**	**125µm > D> 63µm**	**D>125µm**
1	Silica	15g	15g	15g
2	Zeolite	15g	15g	15g
3	Activated Carbon	15g	15g	15g

Figure	Observation/ Explanation
 Figure 14: Grinding Process	- The purpose of grinding is to crunch the granular sample into smaller particle, powder form. - There are total of 9 samples which are required in this experiment. - All of the 9 samples different in particle sizes and particle types.
 Figure 15: Sieving Process	- This project requires 2 different siever with pore sizes of **63µm and 125µm.** - Resulted from this process, all of the powder sample will be segregate following the particle diameter of **D < 63µm, 125µm > D> 63µm an D>125µm**
 Figure 16: Oven for Drying Purpose	- Through out this project, oven is set for 30°C and 120°C. - 30°C is used for drying and removal of moisture trapped in sample. - While 120°C is set for the usage of drying precursors in the following stage.

4.1.2 Stage 2: Fabrication of ordered uniform porous carbon

Chemicals:

a. Sucrose
b. Sodium Hydroxide Solution (4 moles/L)
c. Sulphuric Acid (4 moles/L)

Equipment

a. Oven (120°C)
b. Furnace (650 °C and 850 °C)
c. Electronic weigher (Company: Mettler Toledo)

Apparatus

a. Beaker (500ml, 140ml, 100ml, 80ml, 50ml)
b. Measuring cylinder (25ml)
c. Crucible (50ml)
d. Dilution flask (500ml)

Preparation of Sodium Hydroxide Solution, NaOH (4 moles/L)

- Calculation of NaOH solid to be diluted in 1L of distilled water

$$\text{Molality, M} = \frac{\text{Number of Moles Solute}}{\text{Volute of Solution}}$$

$$\frac{4\text{ moles}}{\text{L}} = \frac{\text{Mass/ MW of Solute}}{1\text{L}}$$

$$\text{Mass/ MW of Solute} = 4\text{ moles}$$

$$\text{Mass} = 4\text{ moles} \times 40\text{ g/moles}$$

$$\text{Mass} = 160\text{ g}$$

Note:

To produce 4mol/L of Sodium Hydroxide Solution, 160g of NaOH solid required to be diluted in 1 L of distilled water.

Therefore, for using only 500ml of distilled water, the total mass that required to be diluted in that amount volume of water is:

$$\frac{160\text{ g NaOH}}{1\text{L}} = \frac{X\text{ g NaOH}}{500\text{ml}}$$

$$X\text{ g NaOH} = 80\text{g}$$

Preparation of Sulphuric acid, H_2SO_4 (4mol/L)

- Given the original purity of H2SO4 is 97%-95%
- Let the purity of H_2SO_4 is in term of wt%/wt%

$$\%\text{ Purity} = \frac{\text{Weight of solute}}{\text{Weight of solute + Weight of solvent}} \times 100\%$$

$$96\% = \frac{\text{Weight of pure } H_2SO_4}{\text{Weight of } H_2SO_4 + \text{Weight of Distilled water}} \times 100\%$$

$$0.96 = \frac{\text{Weight of pure } H_2SO_4}{\text{Weight of } H_2SO_4 + \text{Weight of Distilled water}}$$

Let A = Weight of pure H_2SO_4

Let B = Weight of Distilled water

$$0.96 = \frac{\text{Weight of pure } H_2SO_4}{\text{Weight of } H_2SO_4 + \text{Weight of Distilled water}}$$

$$0.96(A+B) = B$$

$$24A = B$$

If we take 5g of 96% of purity of solution, then we will have

$$0.96 = \frac{\text{Weight of pure } H_2SO_4}{5\text{g}}$$

$$A+B = 5\text{g}$$

$$25A = 5\text{g}$$

$$A = 0.2\text{g}$$

$$B = 4.8\text{g}$$

Calculation to obtain and dilute 4mol/L of H_2SO_4

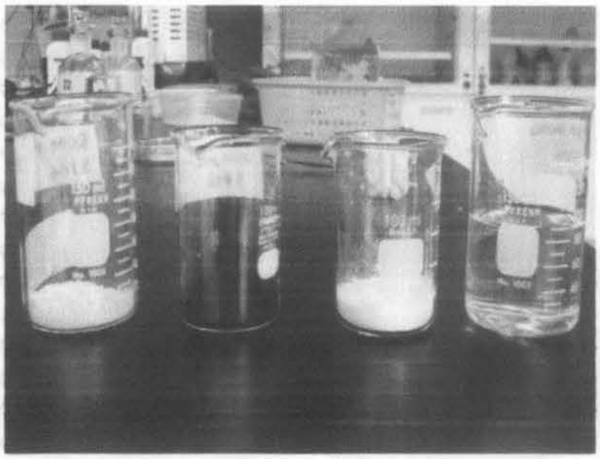
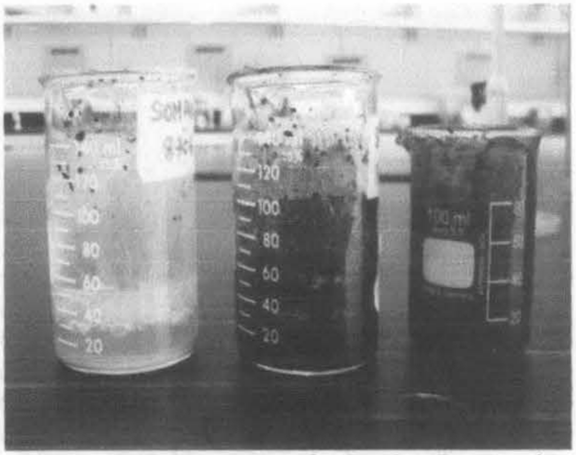
MW for H_2SO_4 is 98g/mol

$$4.8\text{g}/\ 98\text{g/mol} = 0.049 \text{ mol } H_2SO_4$$

$$4\text{mol/L of } H_2SO_4 = \frac{0.049 \text{ mol } H_2SO_4}{\text{X L of distilled water}}$$

$$\text{X L of distilled water} = 0.012\text{L} = 12\text{g}$$

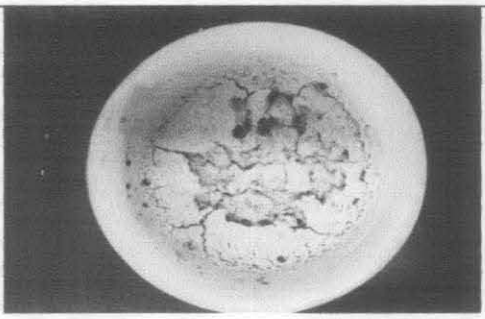
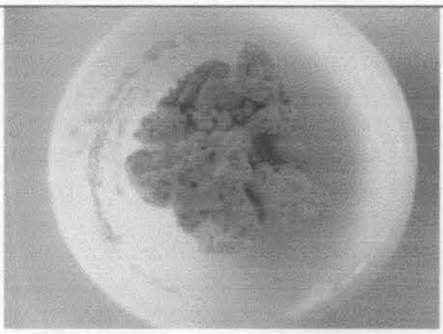
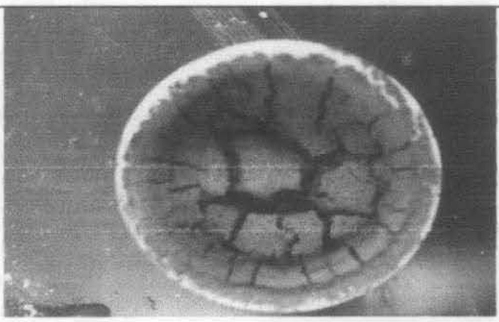
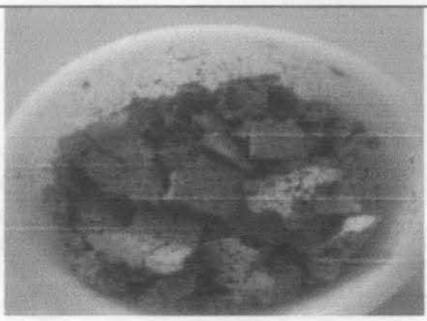
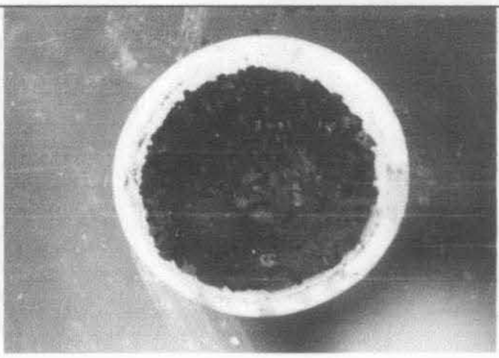
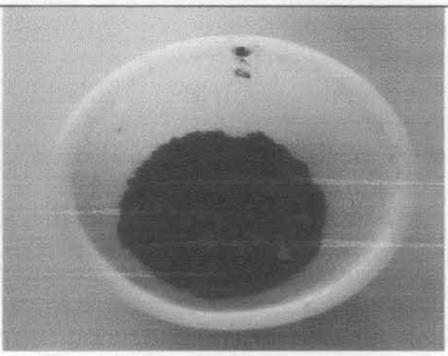
Therefore, in order to obtain 4mol/L from 96% purity of H_2SO_4, for each 5g of 96% purity H_2SO_4, we need to dilute will 12ml or 12 g of distilled water.

Figure	Observation
 Figure 17: Silica Powder, Activated Carbon Powder and Zeolite powder With Carbon Precursor	– Preparation of silica, activated carbon, zeolite and carbon precursor before proceeding with the mixing process.
 Figure 18 : Drop By Drop of Carbon Precursor Added to Zeolite Powder	– It is important to add the carbon precursor solution drop by drop to the sample so that the precursor would be able to penetrate into the pores of the sample uniformly.
 Figure 19: 2 Hours Run for Drying Process in 120°C	– From the figure, dark brown indicates the carbonization of carbon precursor. This drying process is done on stages basis to ensure uniform carbonization of carbon precursor in powder sample.

Procedures: (Experiment 1, Part C)

1. After 6 hours of drying process, all of the samples have to be slowly heated to 650°C and the temperature for is maintained for 7hrs.
2. The samples are cooled down for minimum of 12 hrs before it is proceed with second heating to 800°C. After carbonization of 800°C, the sample is left for self cooling in desiccator for 12 hours.
3. Sodium Hydroxide Solution, NaOH 4 moles/L are used to etch the inorganic template. Etching process is the final step of porous carbon fabrication process.
4. Porous Carbons fabricated are used for further process such as characterization and gas separation testing.

Table 11: Comparison of Sample Heated at 650 °C and 850°C

	Heating at 650°C	Heating at 850°C
(Silica) D < 63μm		
(Zeolite) D < 63μm		
(A. Carbon) D < 63μm		

Procedures: Experiment 2, Part A

Note: Experiment 2 is conducted after the characterization of experiment 1's samples.

1.Step 1-5 from experiment 1, part A are repeated. In this section, there will be no activated particles are tested. Besides that, the particle ranges sizes are narrowed to diameter, D <63 μm and diameter, D > 125 μm.

(Please refer "Discussion" section for further clarification)

Table 12: Inorganic Templates Required in Experiment 2

No	Inorganic Template / Particle Diameter	D < 63μm	D>125μm
1	Silica	15g	15g
2	Zeolite	15g	15g

Procedures: Experiment 2, Part B

1. With the new samples obtained, same procedure from Experiment 1, Part B, and (Step1-10) is repeated.

Procedures: Experiment 2, Part C

1. Step 1 to 4 of Experiment 1 Part C is repeated with different carbonization temperature (a.800°C b.1000°C)

4.1.3 Stage 3: Characterization of fabricated order uniform porous carbon

Note:

Results for Experiment 1, included XRD and SEM, are attached in the **Reference** section. Figure 32 to figure 40are the XRD results, while Figure 41 to figure 56 are the SEM results.

Analysis on Experiment 1(XRD result):

Table 13: XRD Result for Porous Carbon (Peak Length) and Particle Exists in the Porous Carbon (Experiment 1)

Template Material Diameter	Data Interpretation (Particle exists in the Porous Carbon)
Silica D <63μm	• Calcite-$CaCO_3$ (Hexagonal) • Sodium Calcium Aluminum Silicate • Sodium Calcium Oxide Silicate Hydrate
Silica 63μm<D< 125μm	• Portlandite, Syn-$Ca(OH)_2$ (Hexagonal) • Tridymite 2 theta- SiO_2 • Silicon Oxide-SiO_2
Silica D> 125 μm	• Porlandite, Syn –Ca(OH) • Tridymite- SiO_2 • Sulicon Oxide
Zeolite template D <63μm	• $C_2OH_{16}N_2O_4$ • Tridymin Syn SiO_2
Zeolite 63μm<D< 125μm	• SiO_2
Zeolite D> 125 μm	• Chkalovite $N_2BeSi_2O_6$
Activated Carbon D <63μm	• Sodium Calcium Aluminum Silicate $NaCaAl_3Si_3O_{12}$
Activated Carbon 63μm<D< 125μm	• Sodium Aluminum Silicate Gallium • Rubidium Aluminum Silicate Hydrate
Activated Carbon D> 125 μm	• Thermonatriate, $Na_2CO_3.H_2O$

Table 13 lists the particles exist in the silica, zeolite and activated templated porous carbon. With reference to the figure 32 to figure 40 in appendix sections, it is noticed that even same template material used, the resulted particles are different. The differences are obvious, especially in Activated Carbon Template whereby there is no similarity of particles exists in the template at all. In author opinion, factor such as:

a. There is contamination of chemicals during Porous Carbon fabrication which lead to the existence of unidentified and not involved chemicals such as Rubidium Aluminum Silicate Hydrate (in Activated Carbon Templated Porous Carbon), Chkalovite $N_2BeSi_2O_6$ (in Silica Templated Porous Carbon)
b. Improper drying process. Drying process is important to ensure the crystallization of particles occurred in uniform condition

Moreover, it is observed that, the peak for each samples are different even with the same template materials are used. The peak may indicate new structure formed. The variation of peak length may be affected by improper drying process. The author is still in process of identifying the component of each peak shown upon.

Analysis on Experiment 1 (SEM result):

Table 14: Porous Carbon Particle's Diameter Range (Experiment 1)

Template Particle Diameter	Porous Carbon Particle's Diameter Range
Silica D <63µm	2-5µm
Silica 63µm<D< 125µm	4-5µm
Silica D> 125 µm	2-5µm
Zeolite template D <63µm	1-3µm
Zeolite 63µm<D< 125µm	1-4µm
Zeolite D> 125 µm	4µm
Activated Carbon D <63µm	2-5µm
Activated Carbon 63µm<D< 125µm	1-5µm
Activated Carbon D> 125 µm	3-8µm

Note: Activated Carbon 63µm<D< 125µm is not tested due to unavailability of SEM.

The SEM result for each porous carbon is attached in the appendices section. Shown upon is the size range analyzed from the SEM result. The particles formed in the silica porous carbon has average of 3 µm with good distribution of particles. It goes the same for the Zeolite particles whereby theirs distribution are clearly shown from the result. The particle diameter ranges from 1-4 µm. However, activated carbon templated porous carbon shows poor distribution of particles. The existence of particles is less as their existed and grouped in lager diameter.

Difference between Experiment 1 and Experiment 2:

a. Experiment 1 samples are carbonized under temperature of (two steps method) 1. 650 °C and 800 °C while Experiment 2 samples are carbonized in 800°C and 1000°C.
b. Experiment 1 is to observe whether different range of template sizes will affect the fabricated porous carbon structure properties. While experiment 2 is to validate the some of the experiment 1 results.
c. Experiment 2 is done based on improvement of Experiment 1 method.

XRD result (Experiment 2)

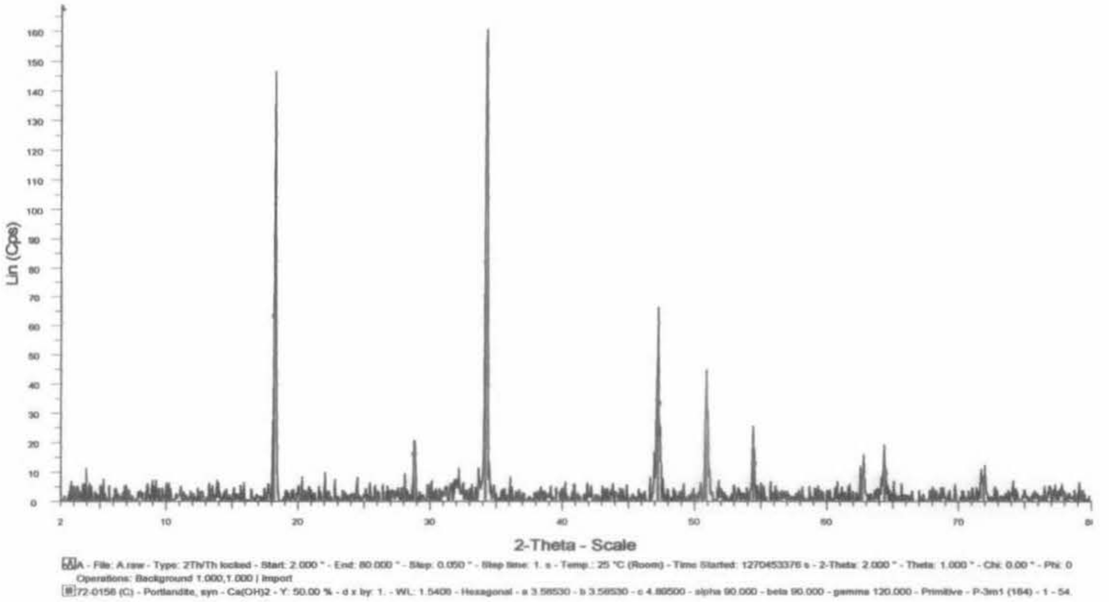


Figure 20: Silica Diameter < 63 µm (Experiment 2)

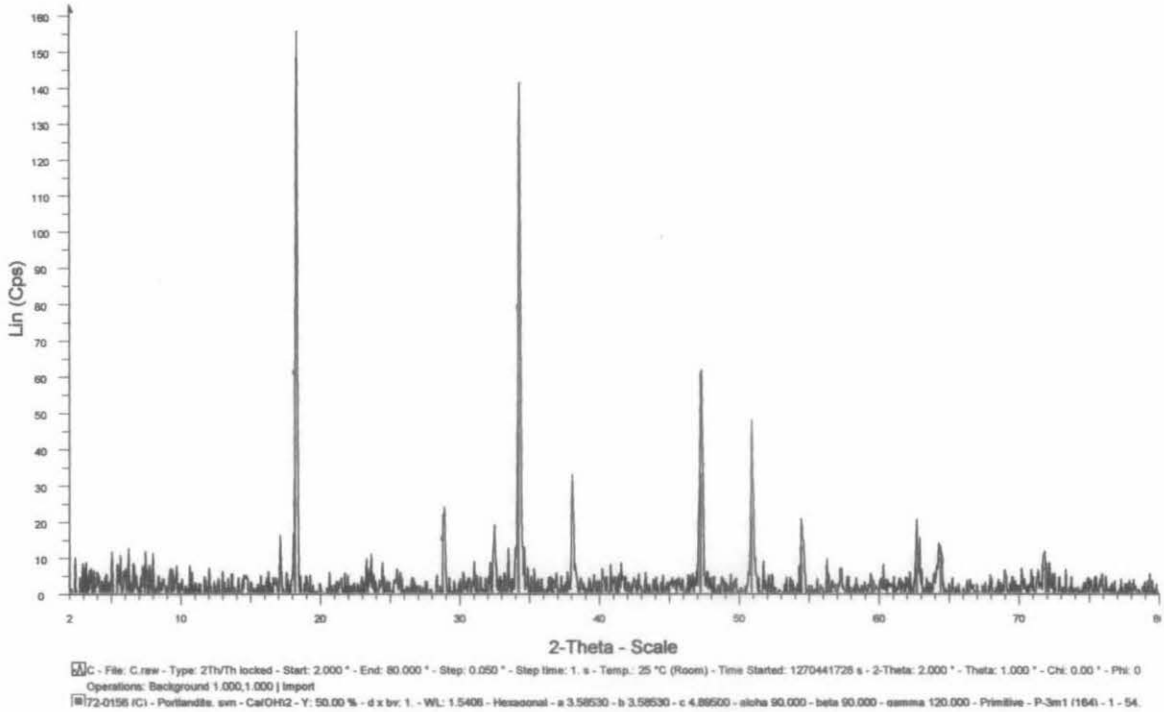


Figure 21: Silica Diameter > 125μm (Experiment 2)

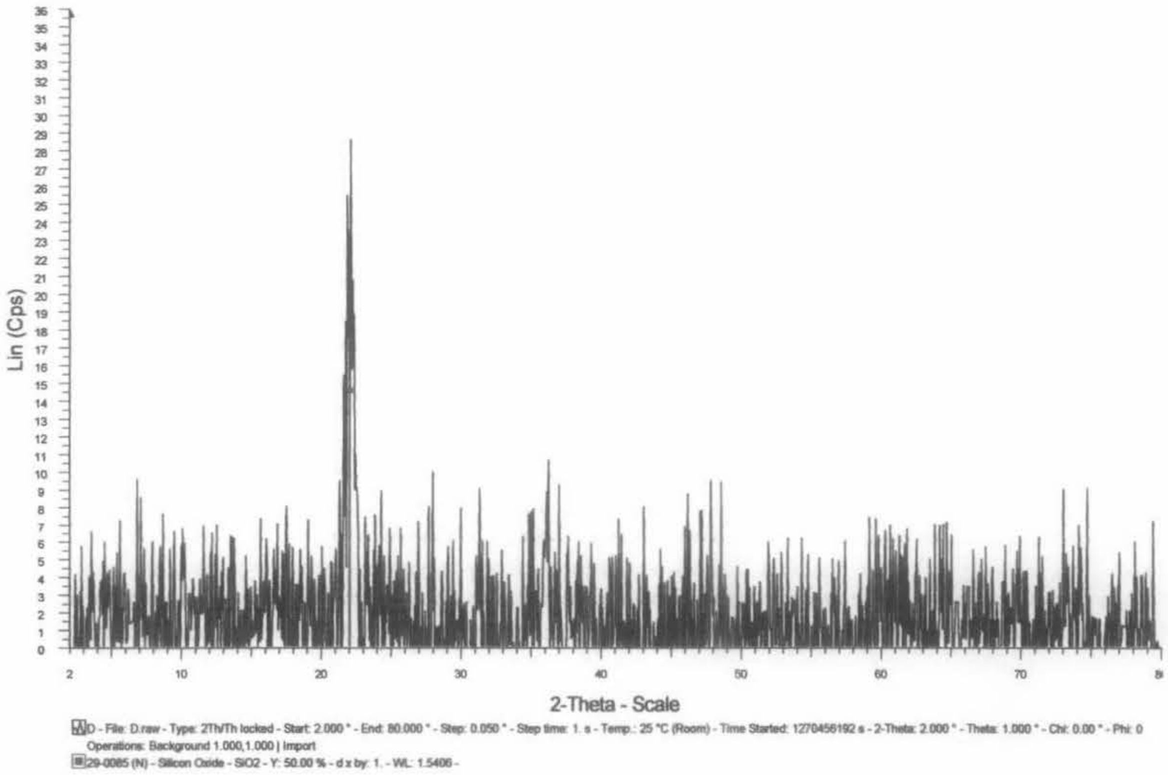


Figure 22: Zeolite Diameter < 63 μm (Experiment 2)

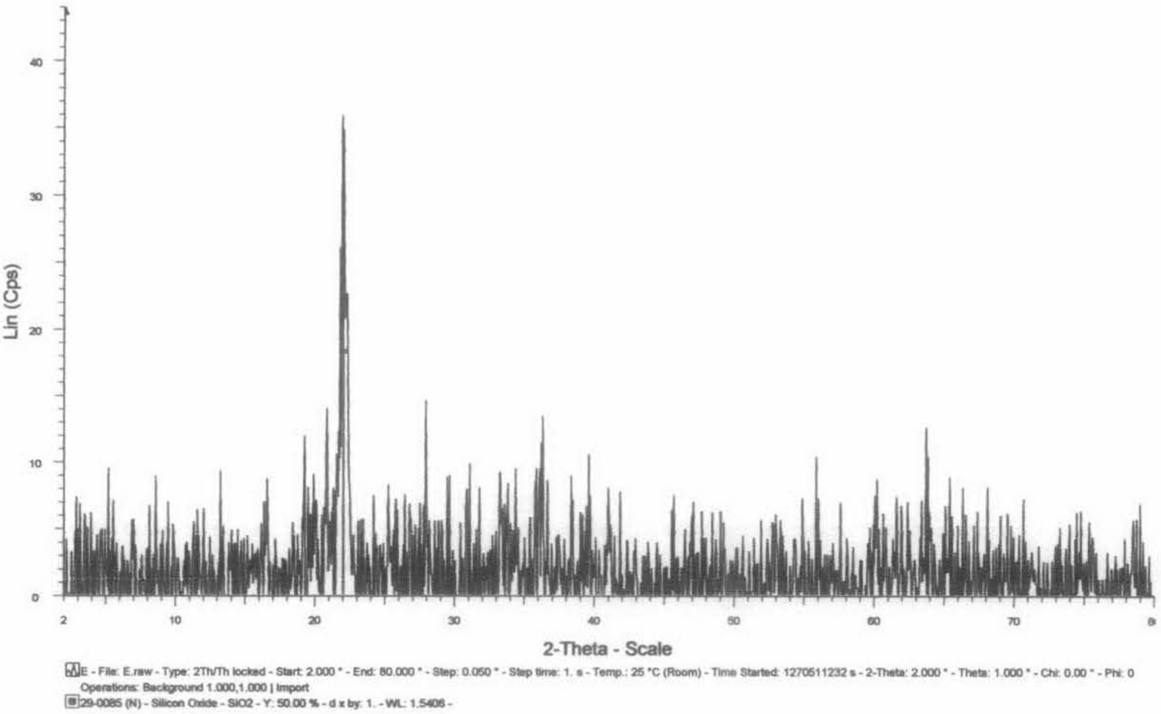


Figure 23: 125μm > Zeolite Diameter > 125μm (Experiment 2)

Analysis on Experiment 2 (XRD result):

In experiment 2, we have basically narrowed the testing samples into 4, originally of 9 samples. Based from the Experiment 1, we could conclude that the **initial template sizes used did not have significant effect on the fabricated porous carbon.** To have confirmation on this, 4 samples, which consists of Silica Diameter < 63 μm, Silica Diameter > 125 μm, Zeolite Diameter < 63 μm, Zeolite Diameter > 125 μm are tested to confirm the structure of the fabricated porous carbon.

Table 15: XRD result for Porous Carbon (Peak Length) and Particle exists in the Porous Carbon (Experiment 2)

Template Material Diameter	Data Interpretation (Particle exists in the Porous Carbon)
Silica D <63μm	• Porlandite,syn- $Ca(OH)_2$
Silica D> 125 μm	• Porlandite,syn- $Ca(OH)_2$
Zeolite D <63μm	• Silicon Oxide- SiO_2
Zeolite D> 125 μm	• Silicon Oxide- SiO_2

Figures 20, 21, 22 and 23 have shown us the consistency of particle existed in the newly formed porous carbon. The existence of Porlandite,Syn -$Ca(OH)_2$ as the particles in silica material templated porous carbon and Silicon Oxide SiO_2 in zeolite templated porous have proven the author the importance of drying process and drying condition in forming uniform particle sizes. Besides that, the results obtained have indicated the essential of being careful, detail, and caution while conducting the experimental work in avoiding contamination of the samples. From the mentioned figures as well, the peak for each samples are same for the same template materials used (different zeolite diameter in the beginning stage). It can be proven trough comparison of figures 20 with 21 and figures 22 with figure 23.

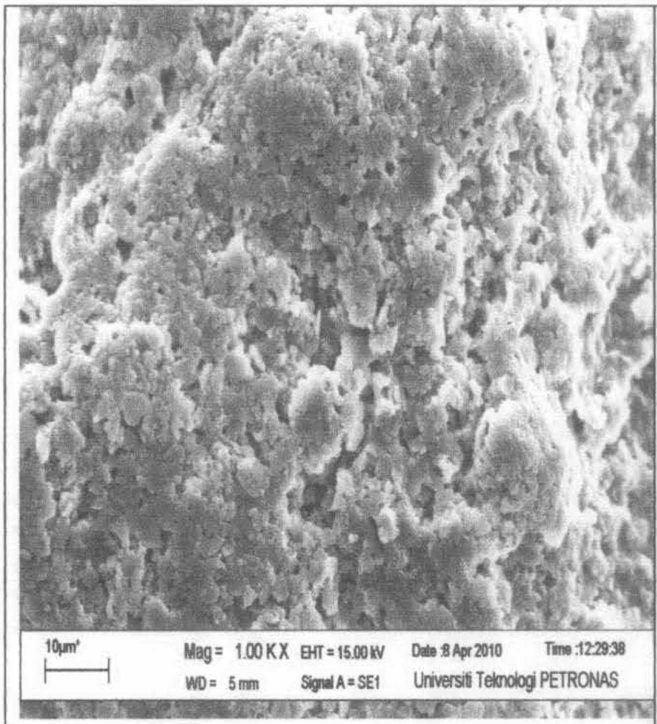


Figure 24: Silica Diameter < 63 µm

(Magnifying Scale: 1000x) (Experiment 2)

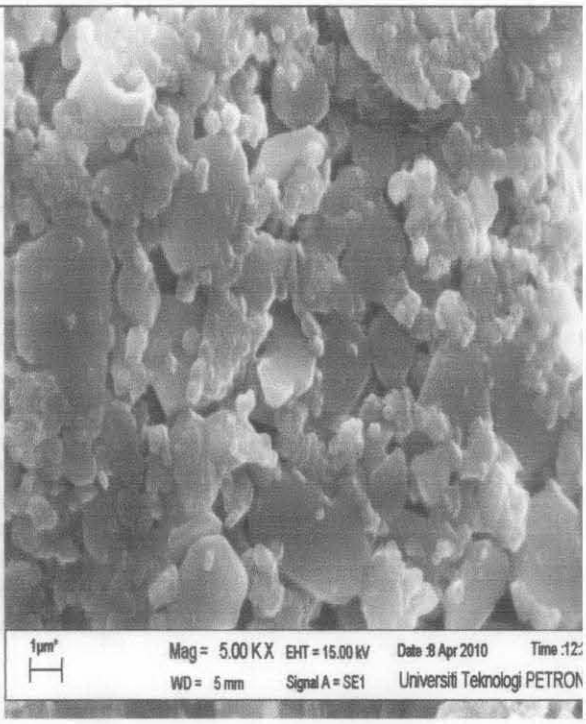


Figure 25: Silica Diameter < 63 µm

(Magnifying Scale: 5000x) (Experiment 2)

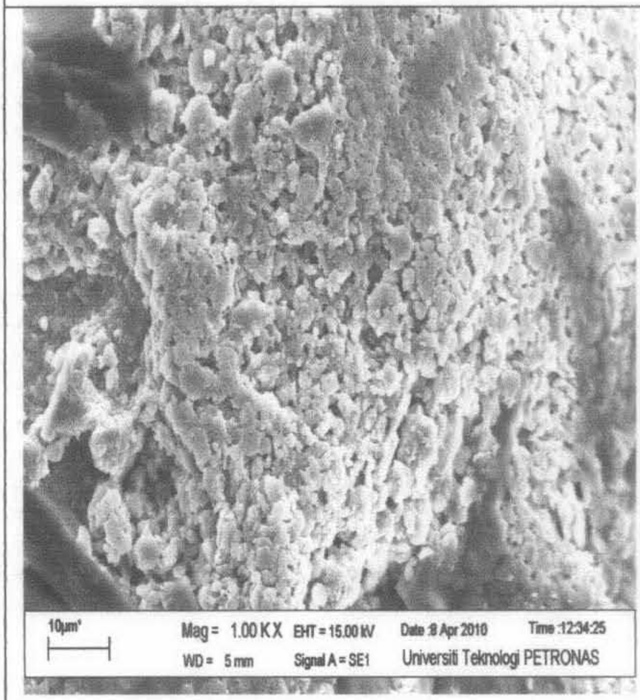


Figure 26: Silica Diameter > 125µm

(Magnifying Scale: 1000x) (Experiment 2)

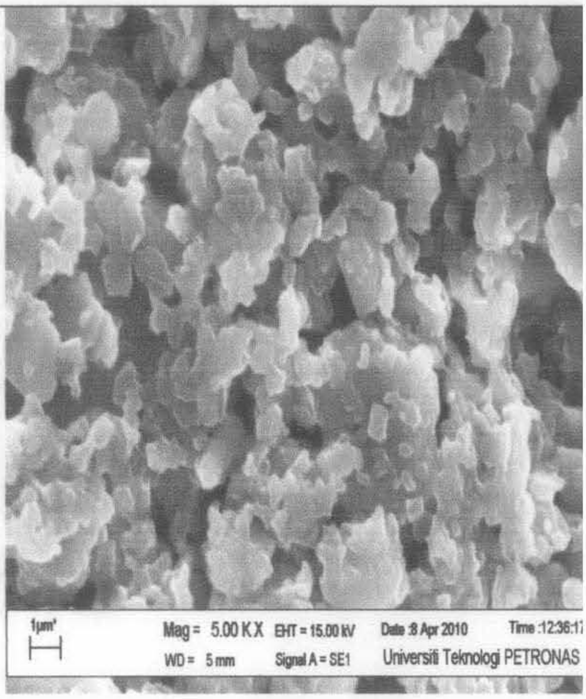


Figure 27: Silica Diameter > 125µm

(Magnifying Scale: 5000x) (Experiment 2)

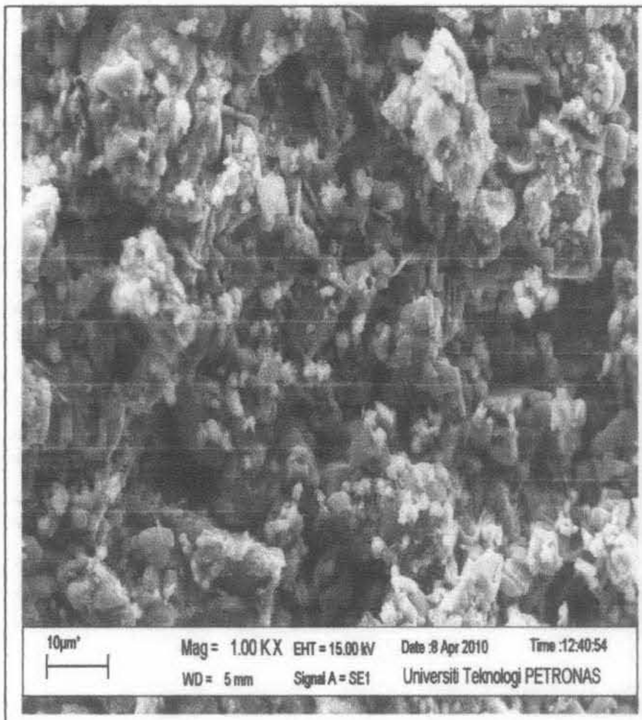


Figure 28: Zeolite Diameter < 63 µm

(Magnifying Scale: 1000x) (Experiment 2)

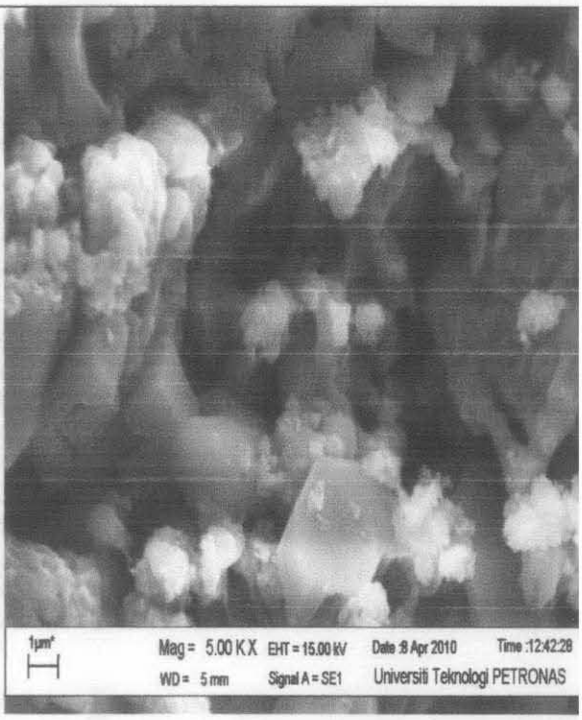


Figure 29: Zeolite Diameter < 63 µm

(Magnifying Scale: 5000x) (Experiment 2)

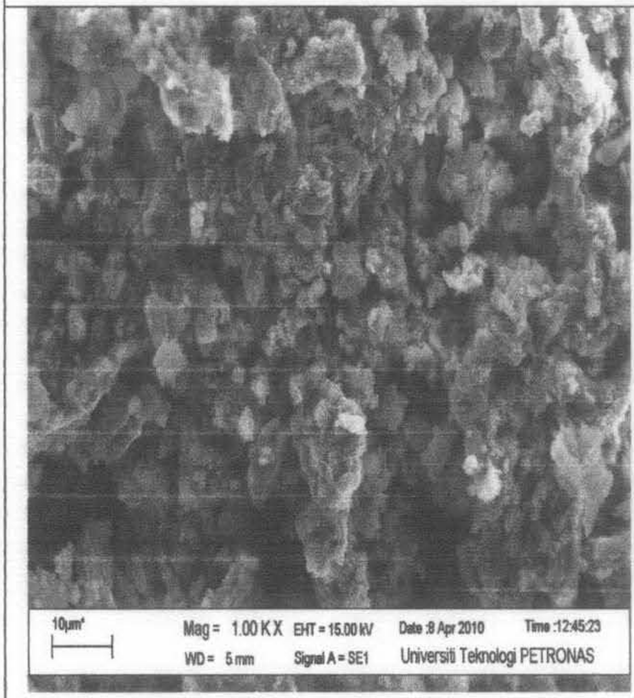


Figure 30: Zeolite Diameter > 125µm

(Magnifying Scale: 1000x) (Experiment 2)

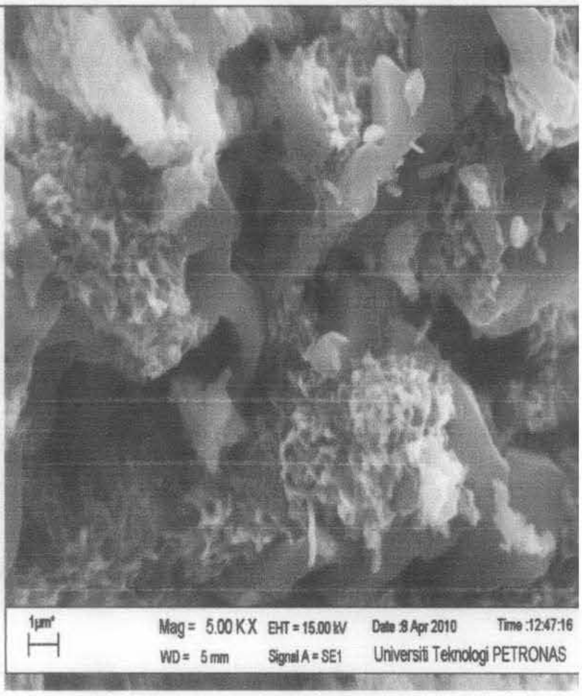


Figure 31: Zeolite Diameter > 125µm

(Magnifying Scale: 1000x) (Experiment 2)

Analysis on Experiment 2 (SEM result):

Figure 24 to Figure 31 are the SEM picture for fabricated porous carbon made of silica, zeolite and porous carbon as the inorganic template. From these pictures, we would be able to determine the pore size and pores distribution pattern as compared to the literature review. In this testing, we are using magnifying scale of 1000 to observe the overall pores distribution and 5000 scale to observe the pore size and its distribution concentrated on a section of the porous carbon. Shown below are the measured particles sizes from figure 24 to figure 31.

Table 16: Porous Carbon Particle's Diameter Range (Experiment 2)

Template Particle Diameter	Porous Carbon Particle's Diameter Range
Silica D <63μm	2-4μm
Silica D> 125 μm	1-3μm
Zeolite template D <63μm	5μm
Zeolite D> 125 μm	3-5μm

4.1.4 Stage 4: Gas separation and analysis

Material:

a. Porous Carbon (Silica templated)

b. Porous Carbon (Zeolite templated)

Chemicals:

c. Solvent Methanol (Still in testing phase)

d. UHU Glue

Equipment

d.Permeable cell

e.Electronic weigher (Company: Mettler Toledo)

f. Pocket Thickness Measurement Gauge Tool

g.Stirrer

Apparatus

e.Flat glass plate (A4 size)

f. Beaker (80ml, 50ml)

Procedures:

1. 2g of Porous Carbon (Silica Templated) is dissolved in 10ml of solvent. The solvent used here is Methanol.
2. The mixture is left for stirred for 24 hrs.
3. The mixture is then mixed with UHU glue to glue the porous carbon which is existed in heterogeneous with the solvent.
4. The mixture-UHU glue is poured on the glass plate then heated for 50°C to evaporate the solvent used.
5. Resulted would be the membrane sheet.
6. The thickness of the membrane sheet is measured before proceed with the CO_2 gas permeation testing.

Noted: Due to some unavoidable reasons (machine breakdowns), the experimental work which scheduled to be done on 20th April has been rescheduled to be on 25th May 2010. At this moment, while waiting for the permeate cell to be repaired, the author are working on finding the suitable solvent to dissolve the porous carbon. The solvent is important for dissolving the porous carbon so that it breaks into smaller sizes which is easier to be glued for membrane sheet forming. Update would be done in this report right after the result is obtained.

4.2 Discussion

In this section, the author would further explained and discussed on the comparison between XRD and SEM results from Experiment 1 and Experiment 2 as sub division below:

4.2.1 Comparison of XRD Result between Experiment 1 and Experiment 2

With reference to the figure 34 to figure 42 and figure 20 to figure 23, the difference of the XRD result obtained from Experiment 1 and Experiment 2 is really obvious. For an instance, even though the same template material and same procedure are applied for Experiment 1 and Experiment 2, the XRD result for figure 32 to figure 34 (Experiment 1) and figure 20 to figure 21(Experiment 2) formed are not the same. The author predicts both set of the experiment results should be the same as only carbonisation temperature is varied trough out the both experiments. Figure 20 to Figure 21 from experiment 2 have almost the same pattern of XRD result as the template material (Silica) used are the same. It goes the same for figure 22 to figure 23 from Experiment 2 whereby the pattern of XRD results are almost the same (Both using Zeolite as the template). However in Experiment 1, it is observed that all of the XRD results are different even same template of materials are used. There are even different particles exist in the same templated material used. For example in figure 34 and 35, even both are using Zeolite as the template, the resulted particles are Silicon Oxide, Tridymite 2H eta and Portlandite in figure 33 while figures 34 gives particles such as Portlandite, Calate syn and Coesite. The author has earlier hypothesis of drying condition most probably is the key affecter. In order to confirm this condition, the author has run the porous carbon production again (noted as Experiment 2) with much precise, careful and different drying and carbonisation temperature. The produced porous carbon end up having more convincing result as same pattern of result obtained based from same template materials. From here, we could conclude that proper drying condition and carbonisation temperature could affect the properties of the resulted porous carbon.

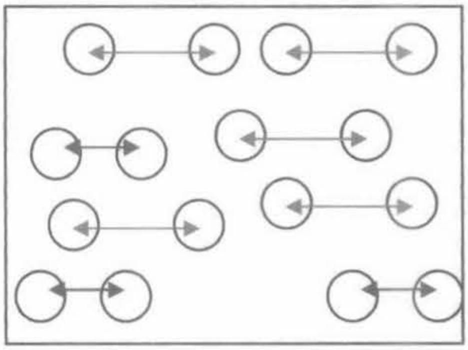


Figure 32: Space between Pores

Noted: Red colour arrow represents shorter space while blue colour represents wider space between the pores.

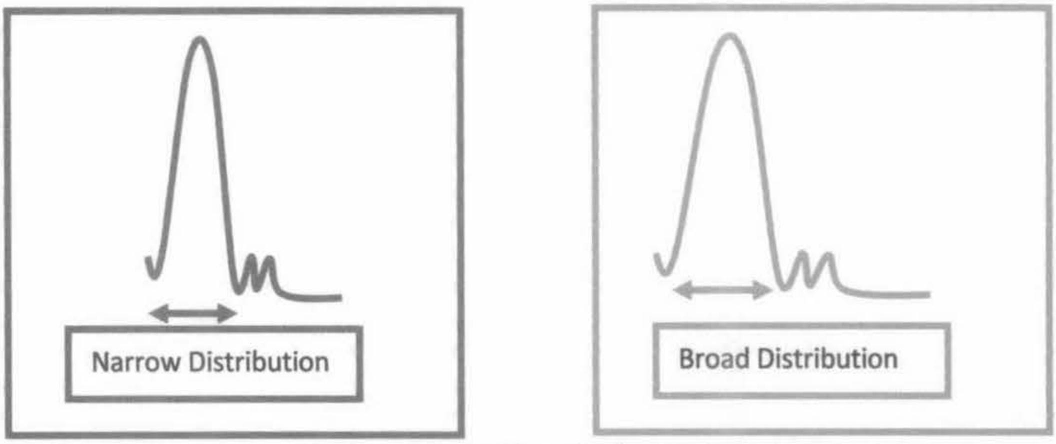


Figure 33: Narrow Distribution and Broad Distribution of Peaks in XRD result

From XRD results of experiment 2, figure 20, 21, 22 and 23, it is observed that the distribution peak for silica is narrower compared to zeolite. Meaning that, the silica pores placed closer to each other as compared to Zeolite. The analogy of pores closer to each other and vice versa is shown in figure 32.

4.2.2 Comparison of SEM Result between Experiment 1 and Experiment 2

There is not much different in term of the particle size range for both Zeolite and Silica templated porous carbon from both experiments. For Silica templated porous carbon, both of the experiments shows the particle size ranges from 2-5μm. While for Zeolite templated, both of the experiments show that the particle size ranges approximately from 1-4μm. Since both of the particle sizes (Silica and Zeolite templated porous carbon) are well distributed and in small sizes, we could say that the porous carbons formed pose higher surface area, hence there would be better possibility of higher total surface area. With more surface area, gas separation process could be done in faster rate, hence affecting the overall selectivity. However, with only depending on SEM result, the order of the pores in the porous carbon whether they are in uniform order or not, is impossible to be determined. For activated carbon templated porous carbon, since the particle sizes range various from 1-8μm as well as the particles are not well distributed, the author eliminates to further investigates on these samples. The author assumes that, with poor particles distribution of the particles, there would be less surface area exposed, hence the gas separation process would be done in slower rate. Slower rate of gas separation will result in lower selectivity value.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In this report, the latest experimental result updated was on 6 May. Generally, the author has managed to cope with 95% of the first objective of this project. The missing 5% is referred to the BET result which could be obtained on 20th May. With BET available, the author would be able to have more information with regard to its porous carbon produced especially the porosity and specific surface area over volume value.

Two main objectives in this project:

a. To fabricate, study and understand ordered uniform porous carbon network
b. To test for CO_2 gas selectivity in gas separation process using porous carbon

Till now, there are three stages of this experiment are done. The author is looking forward to further this experiment on 4th stage by 25th May 2010.

Stage 1: Grinding of inorganic template into desired sizes
Stage 2: Fabrication of order uniform porous carbon network
Stage 3: Characterization of fabricated order uniform porous carbon
Stage 4: Gas separation and analysis

Gas separation stage is really important to determine the application of the fabricated porous carbon. In this gas separation section, the author would like to observe how well the fabricated porous carbon in term of CO_2 gas separation. The result obtained from this section would help the author in understand and relating selectivity of CO_2 in gas separation system. The author manages to achieve only 40% of the second objective due to the break down of permeate cell unit. However, the author is confident that the experimental work could be resumed on 25th May after the unit is repaired.

Overall, the author strongly believes that with only XRD and SEM result, the final determination whether the porous carbon is uniformed ordered could not be made. Even though there is availability of information such as particle's sizes range, peak of each porous carbon fabricated and component exists in the fabricated porous carbon, these data are not sufficient in determining the order of fabricated porous carbon. As mentioned before, with BET characterization, important data such as mesoporous, microporous, porous carbon surface area and porous carbon pore volume could be determined. From this, we could determine the order of the fabricated porous carbon.

5.2 Recommendations

Table 17: Rescheduled Time Frame for Project Progress

Week / **Stages**	**1** (25 -29 Jan)	**2** (1-5 Feb)	**3** (8-12Feb)	**4** (15-19Feb)	**5** (22-26 Feb)	**6** (1-5 Mar)	**7** (8-12Mar)	**8** (15-19Mar)	**9** (22-26Mar)	**10-12** (29Mar-25 May)
i		▨								
ii			▨	▨	▨					
iii						▨	▨	▨		
iv									▨	■

With reference to the table above, the proposed project progress has to be rescheduled due to unavailable of equipment such as BET and Permeate cell. It is a critical stage due to the scheduled was off badly from the original planned. Since the availability of BET machine is out during the peak hour, for future reference, the author suggested that he himself should come out with a backup plan in any experimental work to overcome this type of problem again.

In term of experimental, instead of SEM machine used, the author should look for other equipment which could performed better in zooming and to check for the pores order of porous carbon. Suggested equipment is FESEM. In this project, SEM is used to check for the porous carbon pores order; the result obtained shows only the particle which form on the surface of the porous carbon. More or less, it deviates from the main objective of looking for pores order of porous carbon.

For future work for expansion, improvement and continuation of this project, the author would like to suggest important matters which should take into consideration:

a. Template Material (Size of template material does not matter)
b. Carbon Precursor
c. Carbonization Temperature (two-step method)
d. Carbonization should be done in Nitrogen

For gas separation process, few matters which should look into are:

a. Chemical used to bind porous carbon for the membrane fabrication
b. Besides CO_2 gas, other gases such as SO_2, O_2, N_2 should be tested as well for comparison and verification of the each gases' selectivity

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APPENDICES

XRD result for Experiment 1

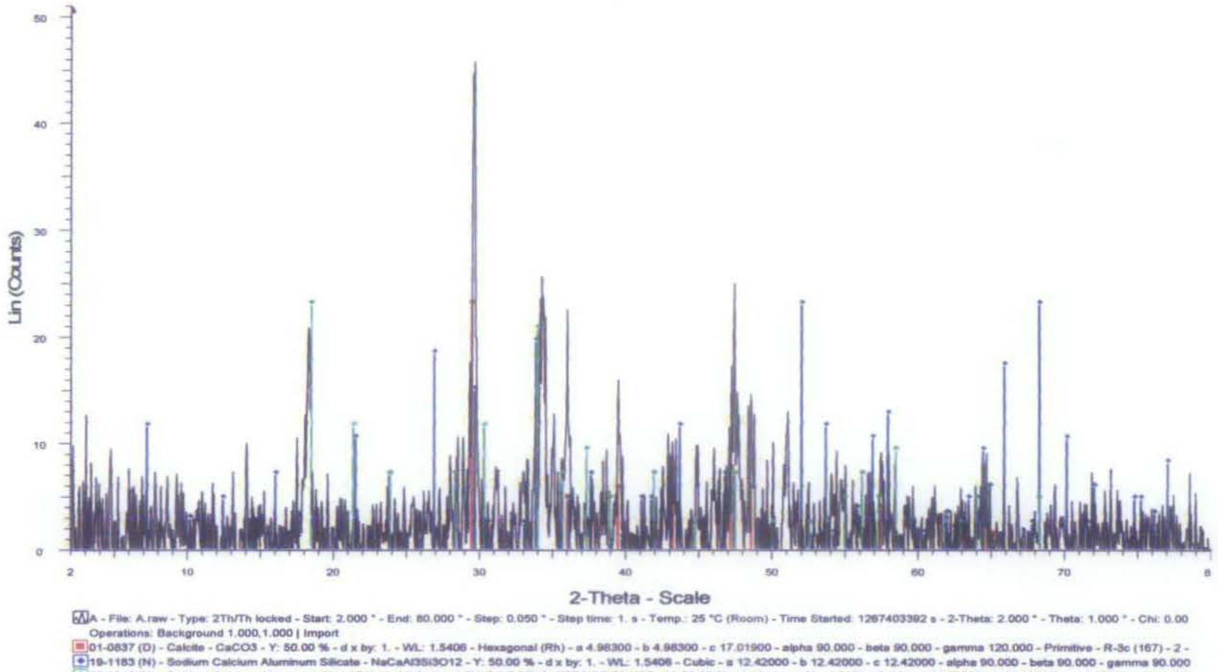

Figure 34: Silica Diameter < 63 µm (Experiment 1)

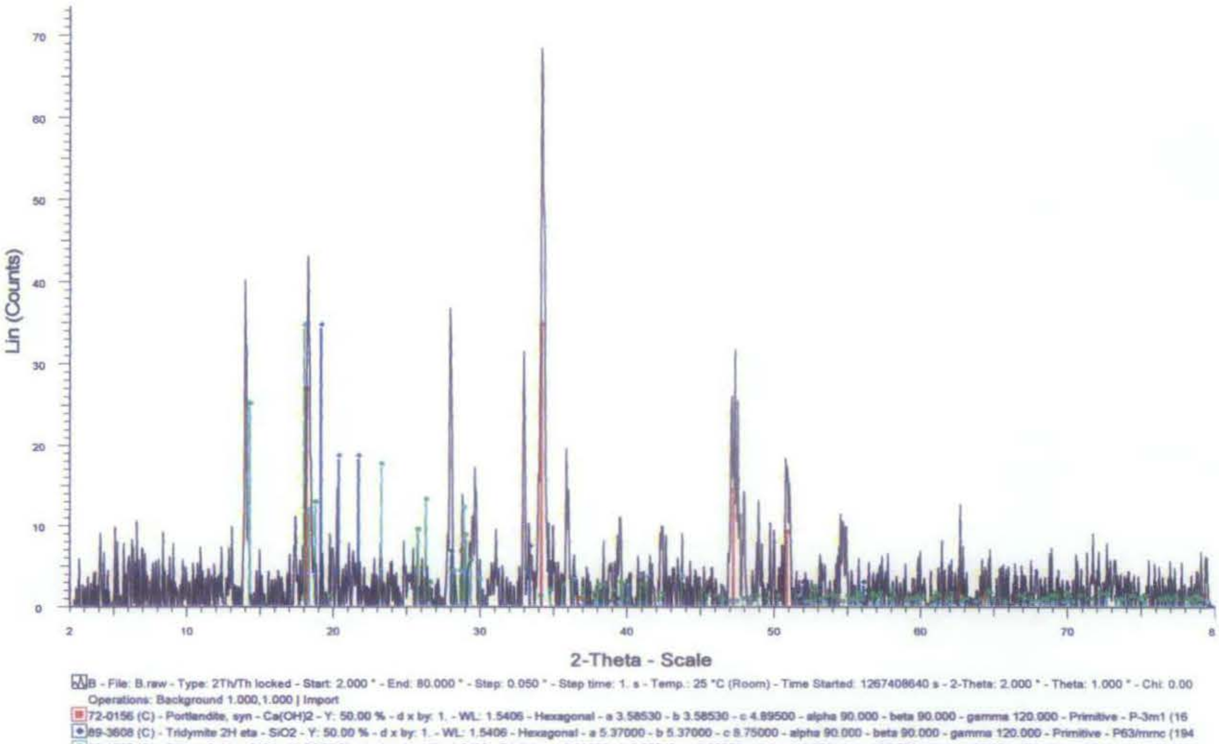

Figure 35: 125µm > Silica Diameter > 63µm (Experiment 1)

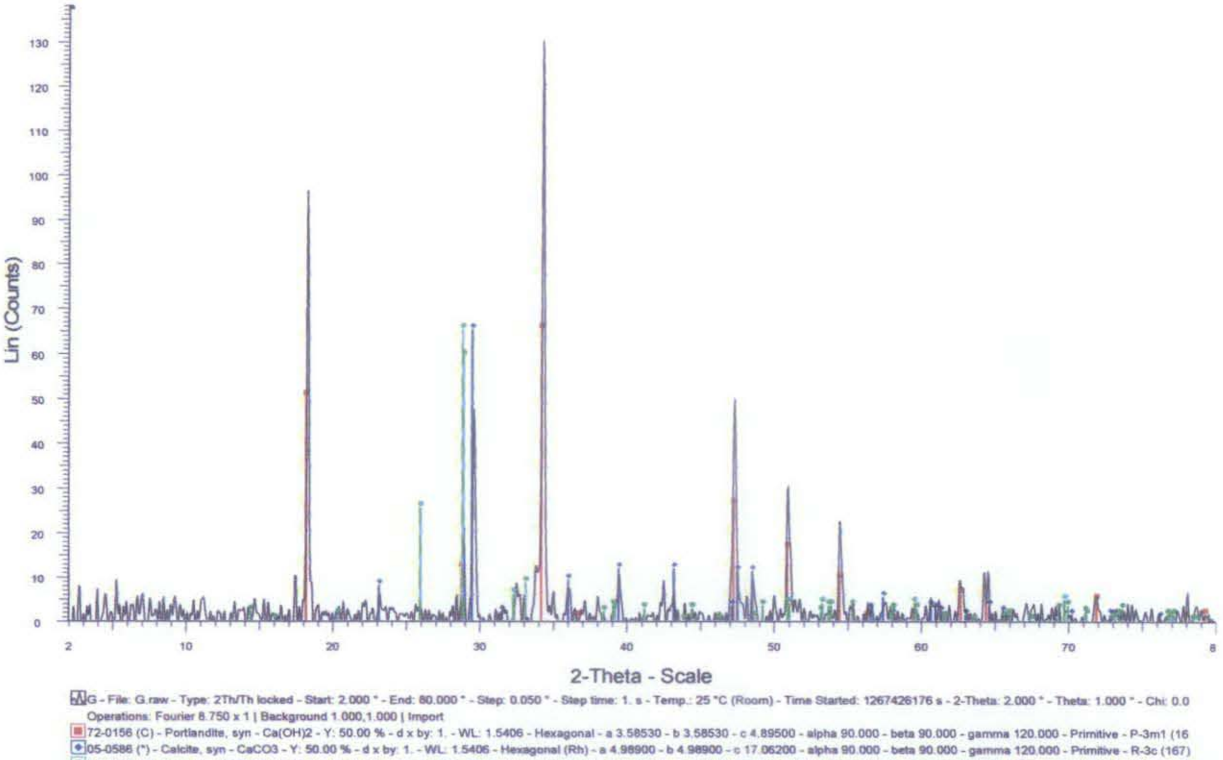

Figure 36: Silica Diameter > 125μm (Experiment 1)

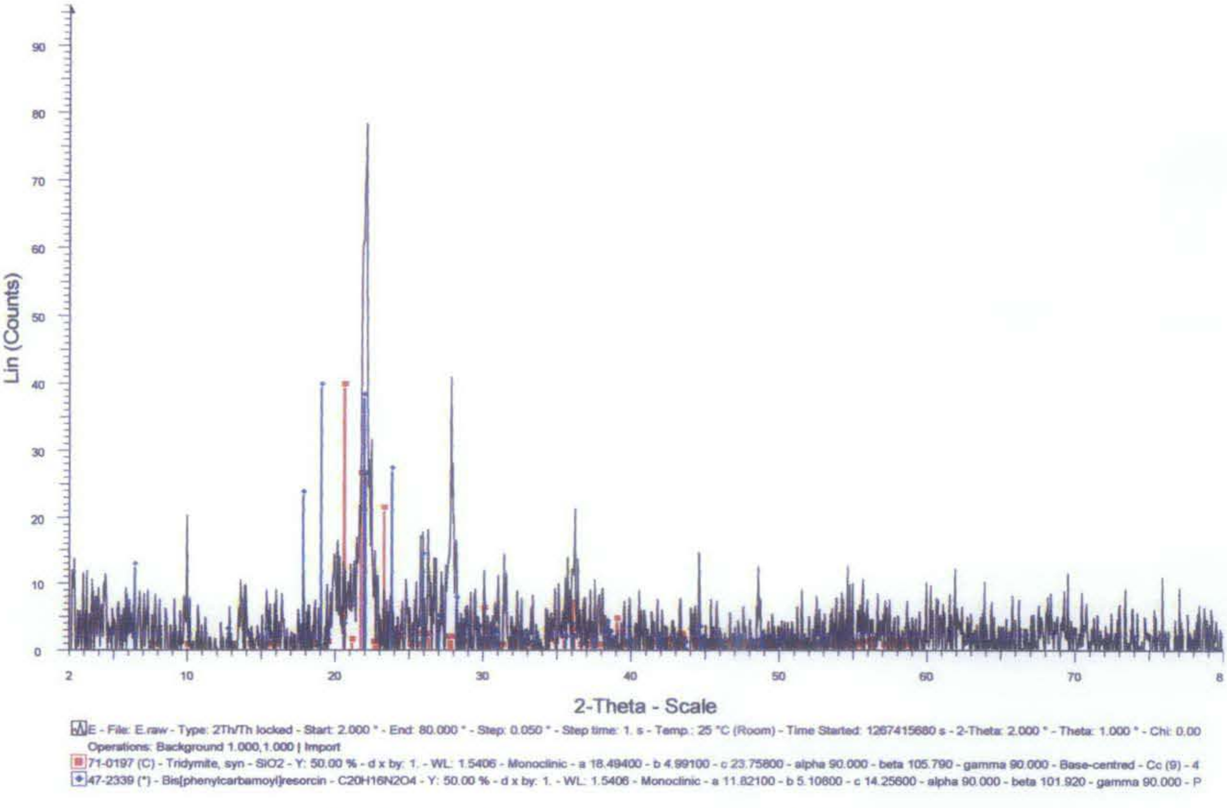

Figure 37: Zeolite Diameter < 63 μm (Experiment 1)

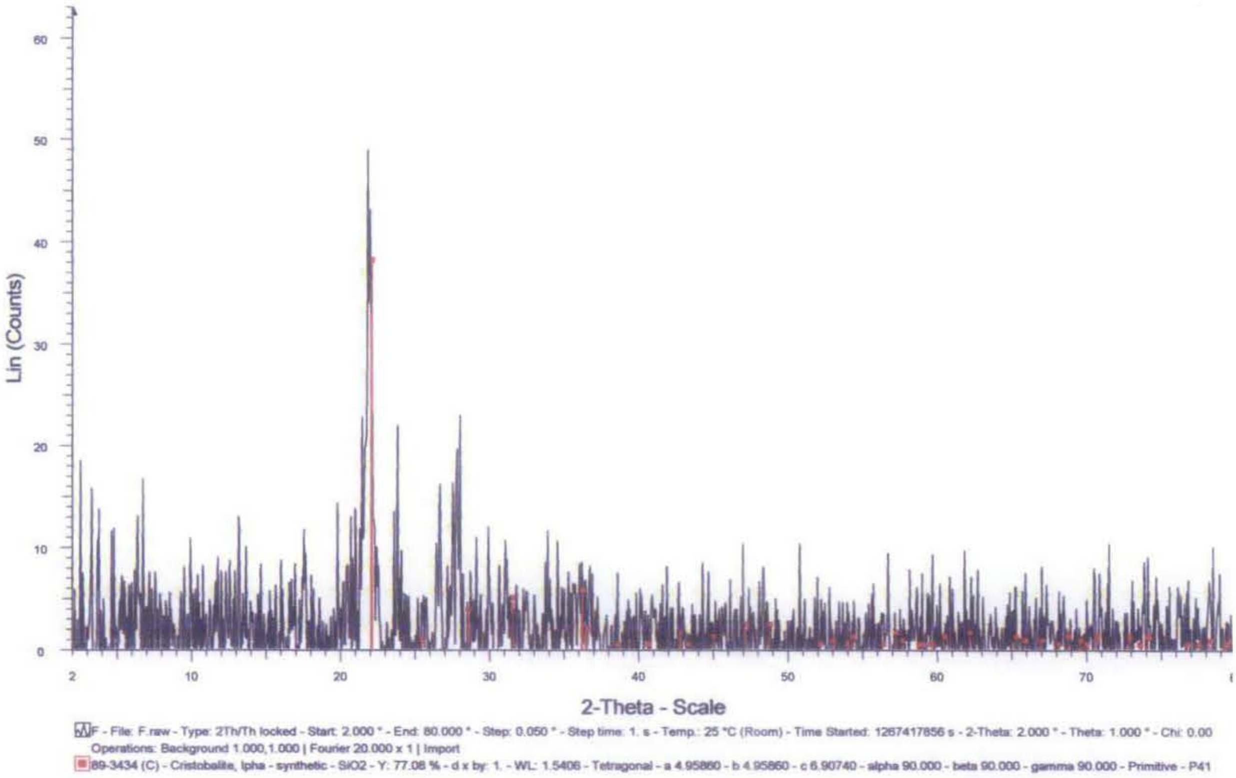

Figure 38: 125μm > Zeolite Diameter > 63μm (Experiment 1)

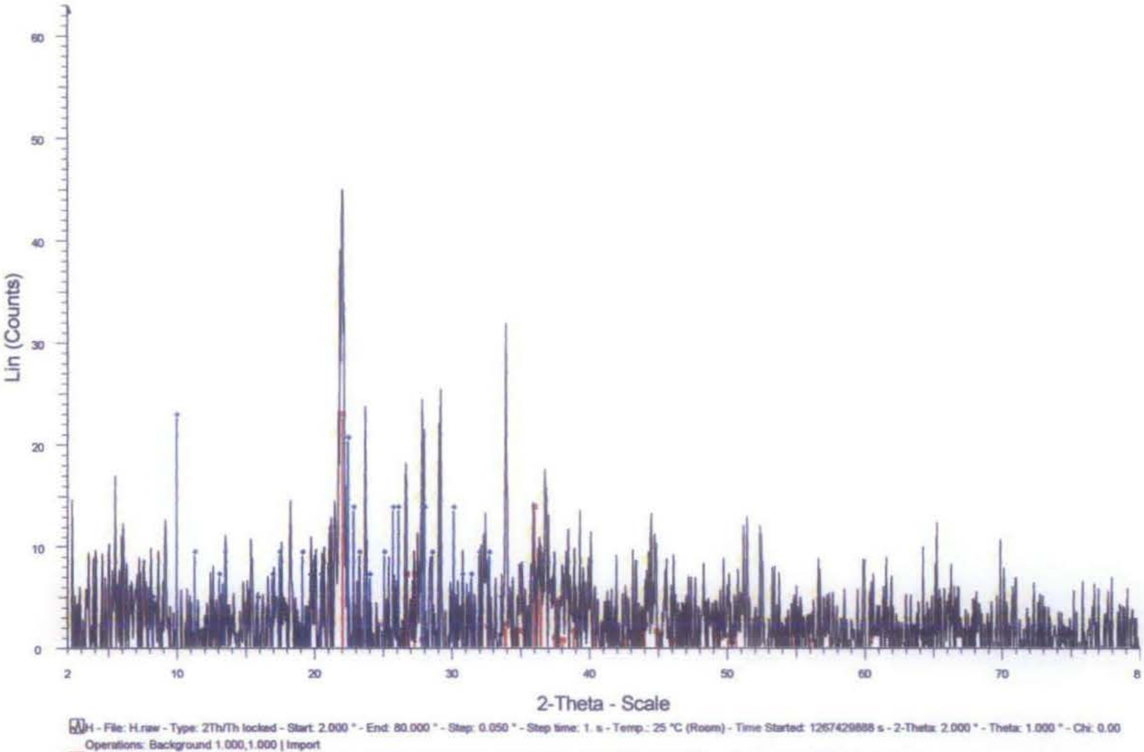

Figure 39: Zeolite Diameter > 125μm (Experiment 1)

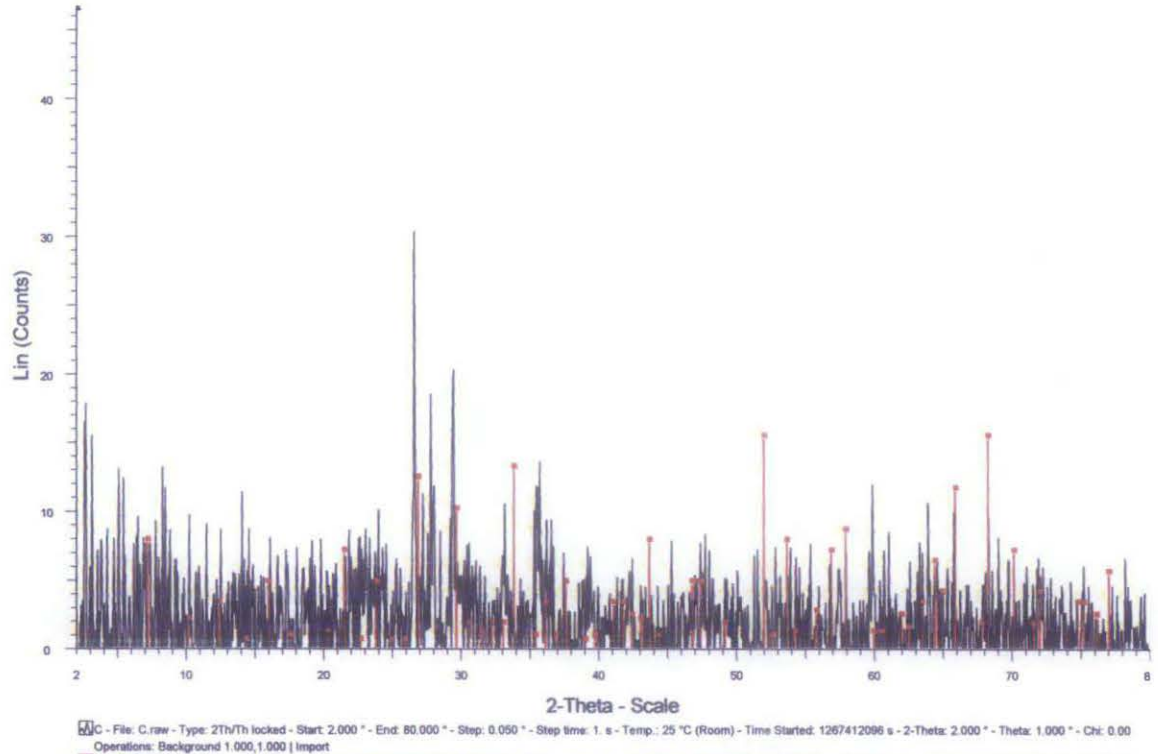

Figure 40: Activated Carbon Diameter < 63 µm (Experiment 1)

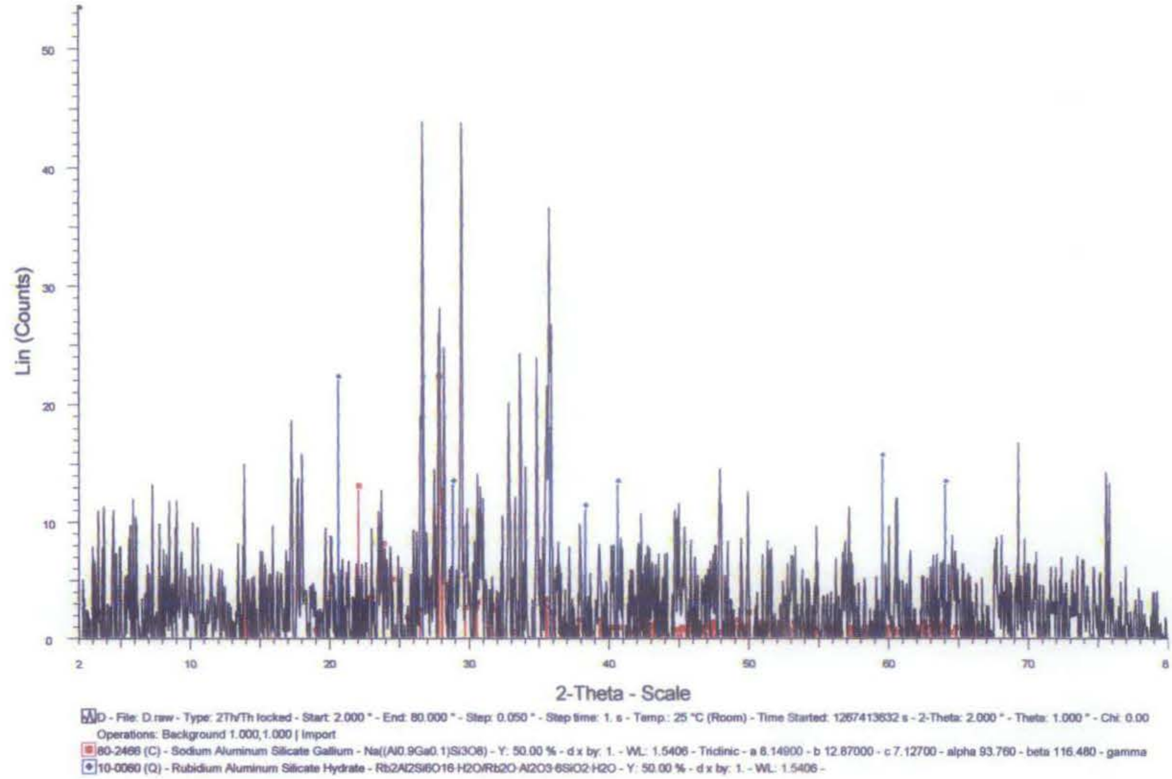

Figure 41: 125µm > Activated Carbon Diameter > 63µm (Experiment 1)

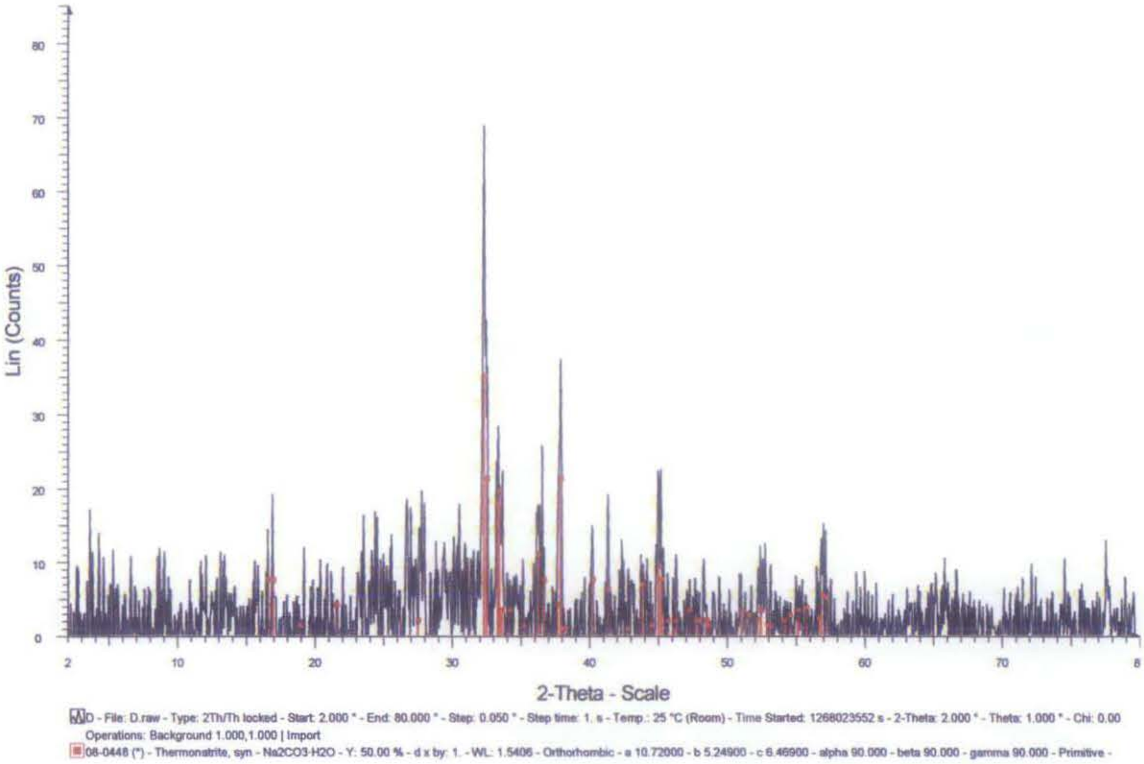

Figure 42: Activated Carbon Diameter > 125 μm (Experiment 1)

SEM result for Experiment 1

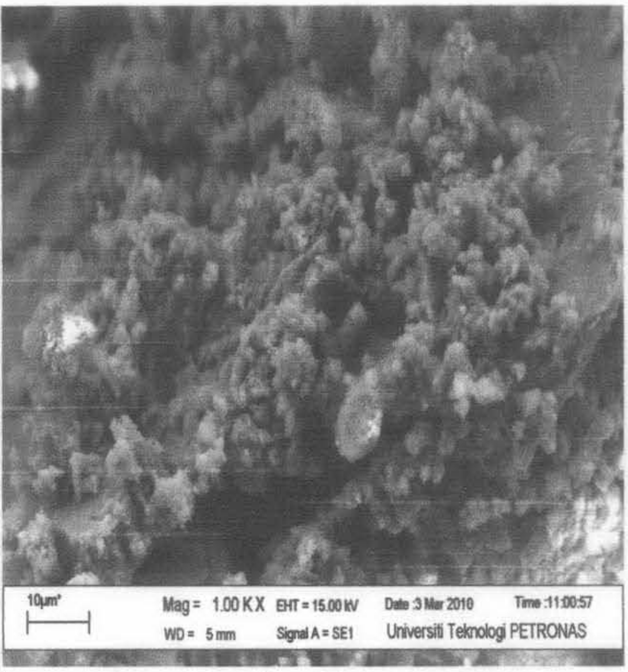

Figure 43: Silica Diameter < 63 µm

(Magnifying Scale: 1000x) (Experiment 1)

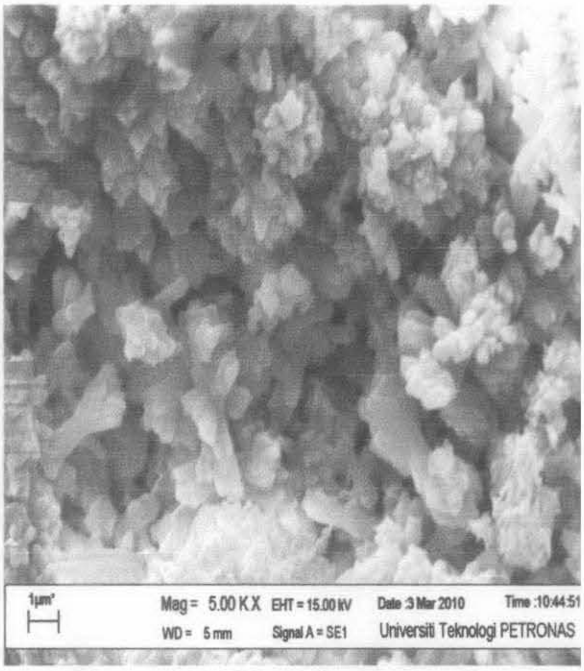

Figure 44: Silica Diameter < 63 µm

(Magnifying Scale: 5000x) (Experiment 1)

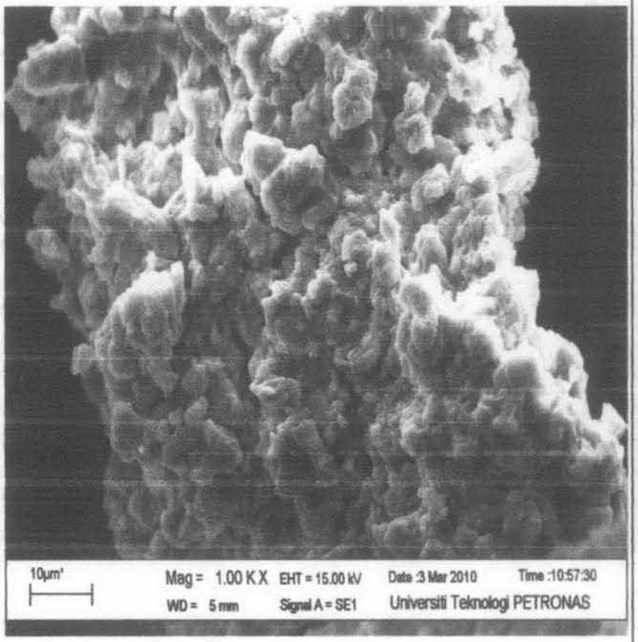

Figure 45:125µm > Silica Diameter > 63µm

(Magnifying Scale: 1000x) (Experiment 1)

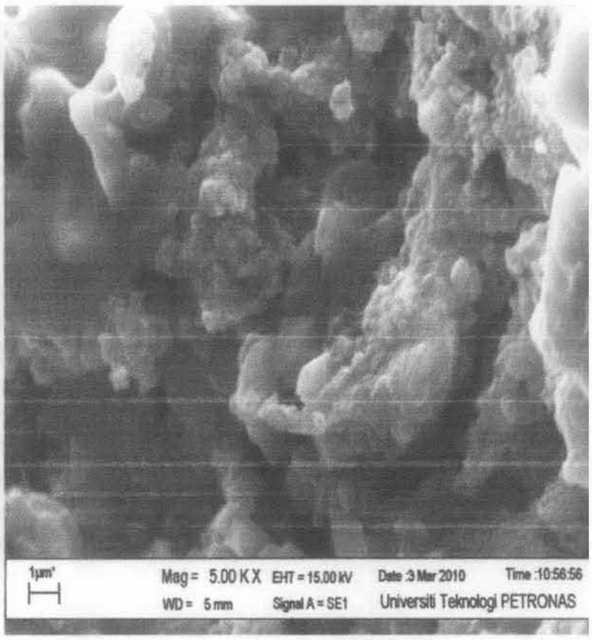

Figure 46: 125µm > Silica Diameter > 63µm

(Magnifying Scale: 5000x) (Experiment 1)

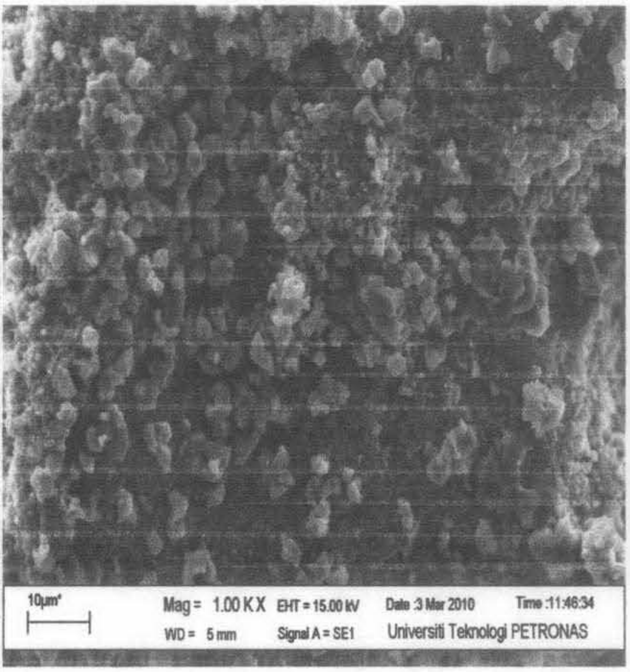

Figure 47 Silica Diameter > 125µm
(Magnifying Scale: 1000x) (Experiment 1)

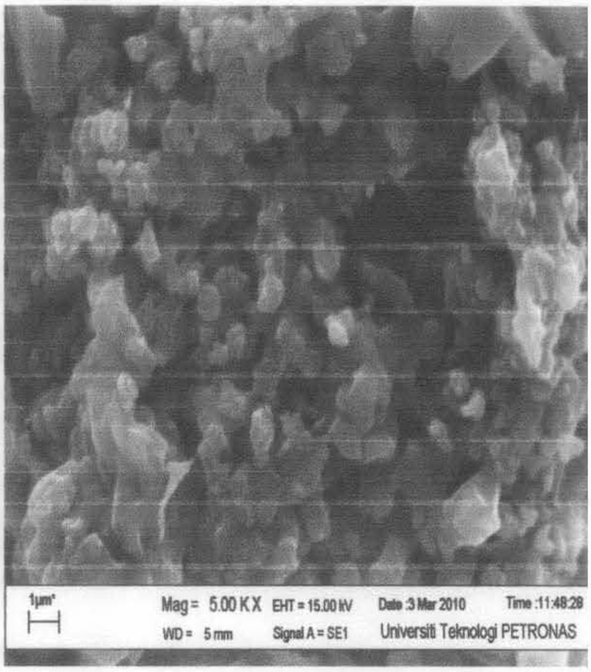

Figure 48: Silica Diameter > 125µm
(Magnifying Scale: 5000x) (Experiment 1)

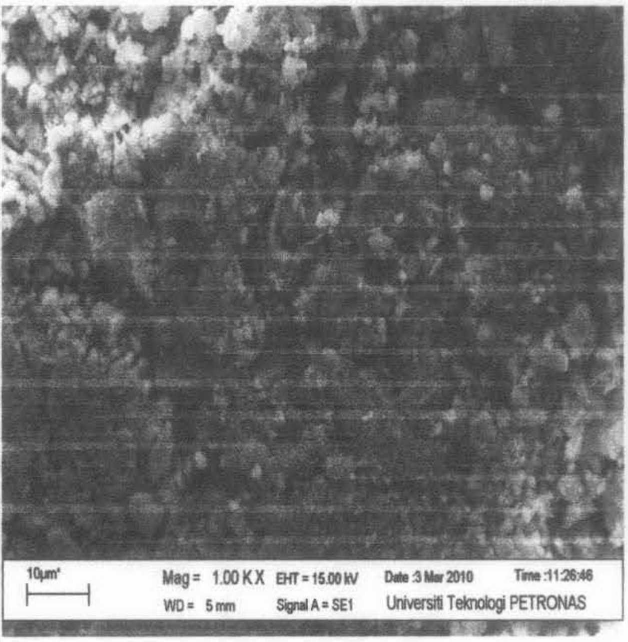

Figure 49 Zeolite Diameter < 63 µm
(Magnifying Scale: 1000x) (Experiment 1)

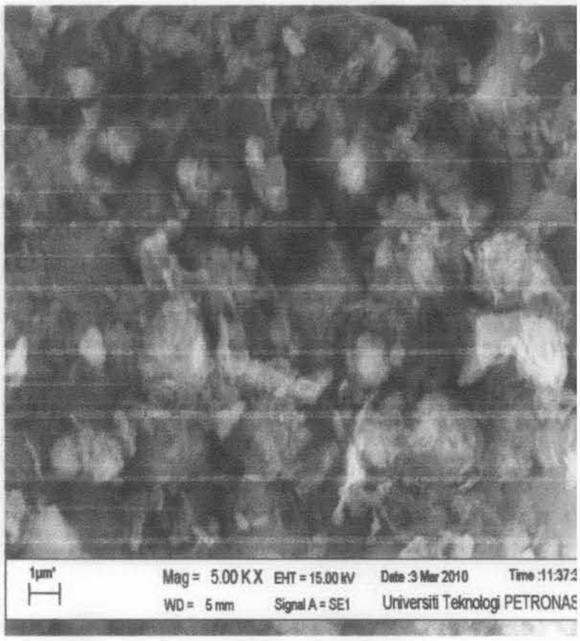

Figure 50: Zeolite Diameter < 63 µm
(Magnifying Scale: 5000x) (Experiment 1)

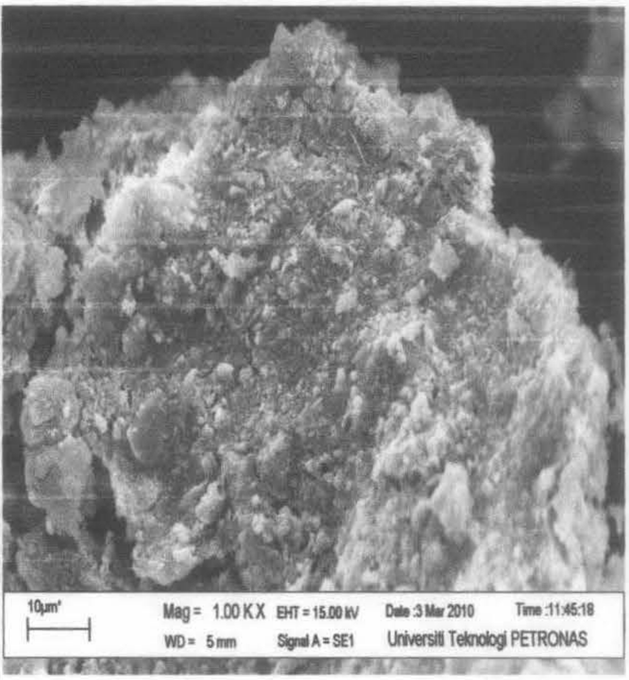

Figure 51: 125µm > Zeolite Diameter > 63µm (Magnifying Scale: 1000x)(Experiment 1)

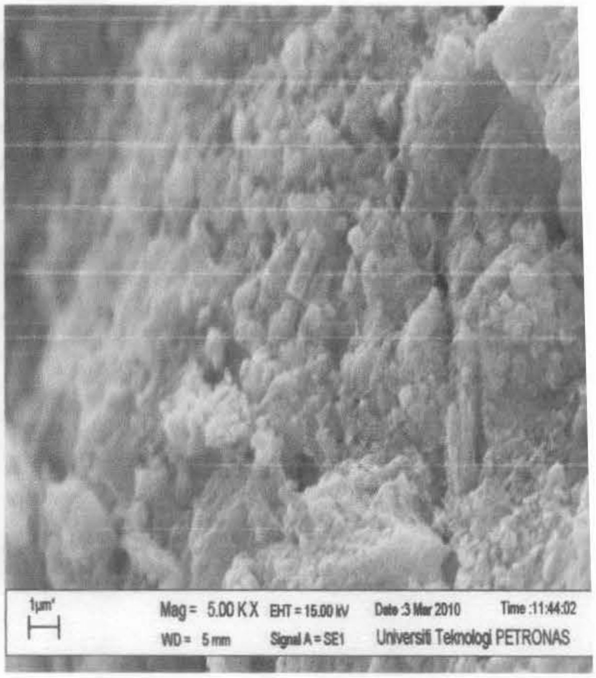

Figure 52: 125µm > Zeolite Diameter > 63µm (Magnifying Scale: 5000x)(Experiment 1)

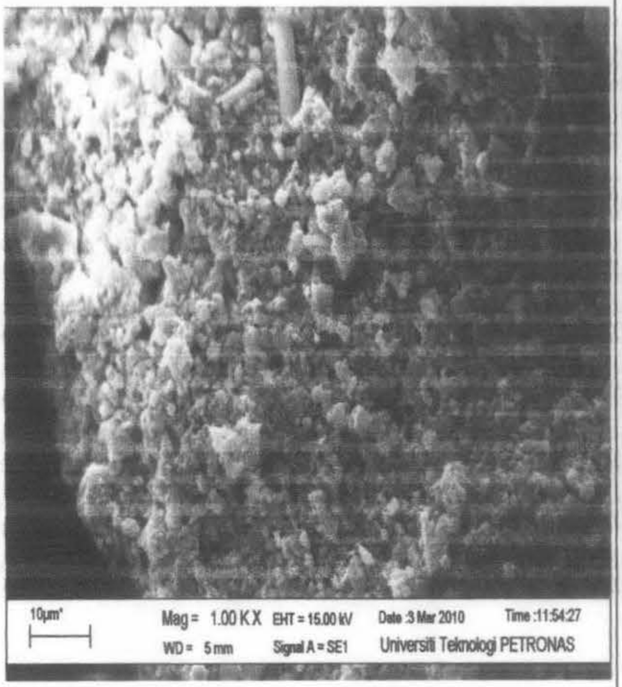

Figure 53: Zeolite Diameter > 125µm (Magnifying Scale: 1000x)(Experiment 1)

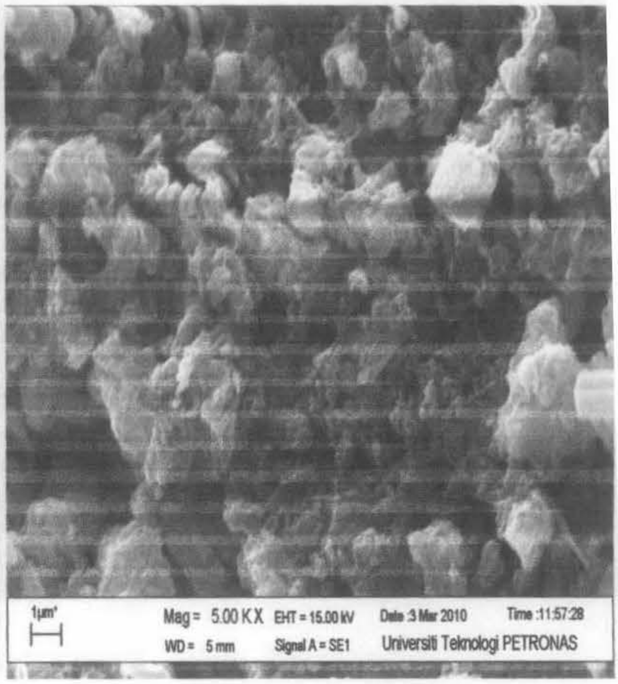

Figure 54: Zeolite Diameter > 125µm (Magnifying Scale: 5000x) (Experiment 1)

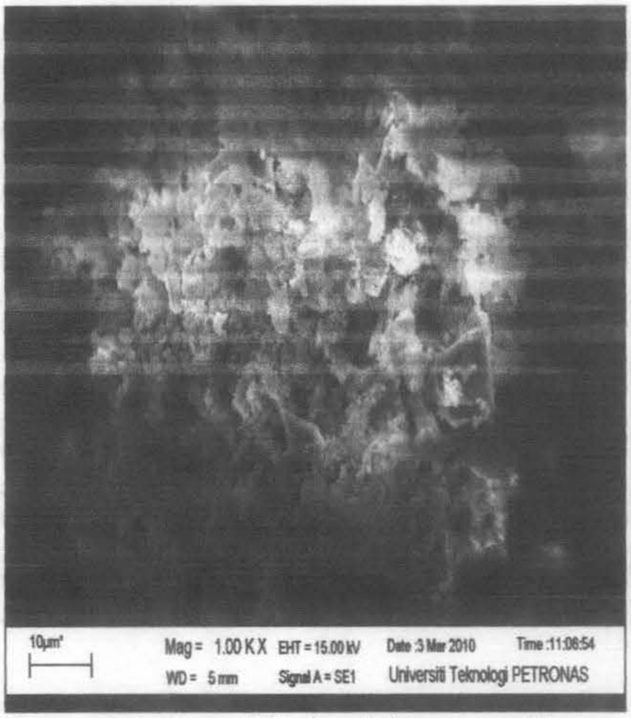

Figure 55 Activated Carbon Diameter < 63µm (Magnifying Scale: 1000x)(Experiment 1)

Figure 56: Activated Carbon Diameter < 63µ

(Magnifying Scale: 5000x) (Experiment 1)

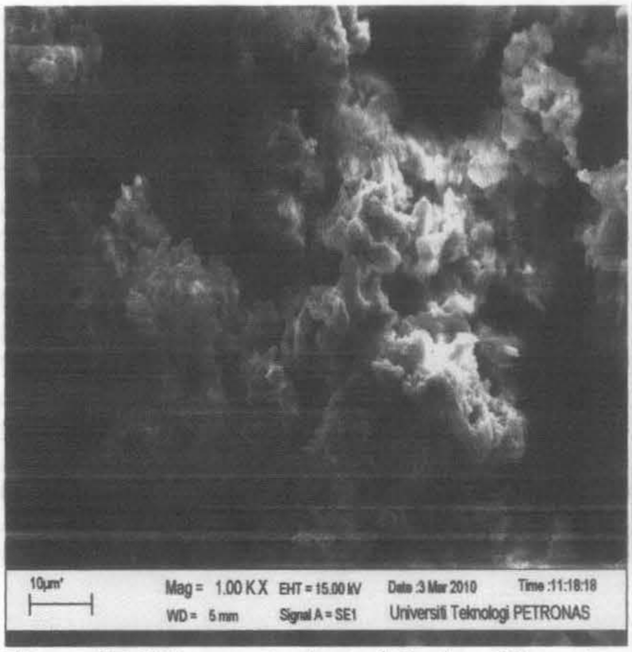

Figure 57:125µm > Activated Carbon Diameter > 63µm (Magnifying Scale: 1000x) (Experiment 1)

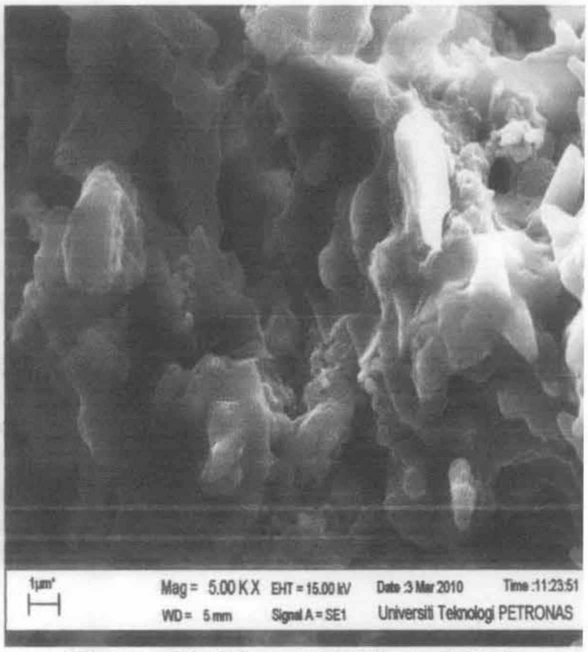

Figure 58:125µm > Activated Carbon Diameter > 63µm (Magnifying Scale: 5000

(Experiment 1)